

SELF-SIZING OF CORRUGATING MEDIUM

Project 3321

**Report One
A Progress Report
to**

MEMBERS OF THE INSTITUTE OF PAPER CHEMISTRY

June 30, 1982

THE INSTITUTE OF PAPER CHEMISTRY

Appleton, Wisconsin

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SUMMARY

A study has been made of the tendency for self-sizing (water resistance, air sizing) to develop in handsheets formed from pulps used in the production of corrugating medium. A survey of corrugating manufacturers indicated that approximately one-half of the companies contacted experience difficulties which are believed to be related to self-sized corrugating medium. Four types of pulps used in producing corrugating medium were examined in the program: a conventional neutral sulfite semichemical (NSSC) pulp, two green liquor semichemical (GLSC) pulps, an alkali-carbonate (AC) pulp, and a recycled fiber pulp. The water absorbency of handsheets formed from these pulps was determined by a waterdrop test by recording both the time required for the drop to penetrate through the sheet and the time required for the drop to be completely absorbed by the sample. The latter measurement received primary emphasis in the program. Handsheets formed from the various pulps were aged at room temperature (23°C) and at 105°C over a range in pH, i.e., 7, 9, and 11. Aging was first carried out in the absence of added chemicals and subsequently in the presence of various additives including rewetting agents, calcium chloride, and sequestering and chelating agents. Results obtained in the absence of additives showed that sheets formed from the green liquor pulps developed self-sizing at the highest rate of all samples tested; waterdrop values of 600+ seconds were obtained in all oven-aged samples within 30 minutes regardless of pH. Aging at room temperature produced similar results in one GLSC pulp and rapidly increasing water resistance in the other GLSC sample. The recycled fiber pulp was least subject to self-sizing followed by the AC pulp. The NSSC pulp occupied an

intermediate position with respect to self-sizing development. Contrary to expectations, little consistency was found in the effect of pH on the development of self-sizing except in the case of the alkali carbonate pulp. The AC pulp showed the highest waterdrop values at pH 11 and the lowest at pH 7 at both aging temperatures. While the trend was consistent in that case, the order was the reverse of that expected.

Addition of anionic wetting agent to the pulp prior to sheet formation improved water absorbency to varying degrees depending upon the pulp and pH. However, the self-sizing effect was not eliminated and, in the case of one GLSC pulp, the rewetting agent provided little or no benefit. Addition of 0.1% of a nonionic surfactant at sheet forming temperatures of 23 and 60°C greatly improved water absorbency in one of the green liquor pulps, possibly to the point of acceptability. In general, increasing the sheet forming temperature of a GLSC pulp in the absence of surfactant reduced the self-sizing effect to the point at 90°C where self-sizing was essentially eliminated. However, since a 90°C forming temperature is an unrealistic condition, emphasis is placed on the results obtained with surfactants at 23 and 60°C.

In other tests, classification of the various pulps to remove fines reduced self-sizing in all but one pulp (GLSC), which was found to retain a high level of solvent extractables and free organic acids after classification. As might be expected, the combination of classification followed by solvent extraction eliminated self-sizing, but this is not a practical alternative to the surfactant treatments described above.

Treatment of GLSC pulp with hydrochloric acid produced significant reductions in self-sizing, presumably due to removal of calcium and magnesium ions. The effect of added calcium chloride was found to depend on pH. Addition of the salt at

pH 7 tended to reduce self-sizing, whereas a detrimental effect was obtained at pH 11 in several cases. The use of sequestering and chelating agents produced some increases in water absorbency in GLSC pulp but, in general, the papers retained some measure of self-sizing under the conditions employed. Thus, in effect, the combination of moderately high forming temperatures and a moderate addition of wetting agent provided the most satisfactory results in this program.

INTRODUCTION

This is Progress Report One on Funded Research Project 3321 entitled "Self-Sizing of Corrugating Medium." This problem was brought to our attention by our Board of Trustees Research Advisory Committee. The problem is described briefly as one involving development of excessive sizing of corrugating medium (resistance to penetration by aqueous liquids) during normal storage of rolls of medium prior to converting in the boxboard plant. Generally no rosin or other internal sizing agent is added to the medium stock. The sizing develops from the natural fatty and resin materials in the pulp. For this reason the problem is sometimes referred to as "self-sizing" or "air-sizing." The resistance to liquid penetration is believed to be responsible for poor fluting of the medium and for inferior adhesion of liner to medium at normal corrugating speeds. Self-sizing is apparently a costly problem to many manufacturers of corrugated board.

A preliminary survey of a number of corrugators led to conflicting opinions about the nature of this problem and methods of coping with it. Some companies said that the judicious use of wetting agent controls the problem but at excessive cost for the surfactant (upward of \$250,000 per year). Others said no adhesion difficulty was encountered, even with hard-sized medium, provided this fact was known before use so that suitable machine adjustments could be made. Still other companies said that self-sizing caused high-low fluting which was then responsible for poor adhesion. These opinions indicated that further input about the problem was needed from experienced people of the industry before formulating the research plan.

A questionnaire designed to determine the nature and importance of the self-sizing of corrugating medium to the industry was mailed to 45 corrugating manufacturers. A copy of this questionnaire is included as Appendix I of this report.

Over 69% of the companies returned completed surveys. A summary of the results is presented in Table I. The responses revealed that approximately one-half of the manufacturers currently experience problems which are considered to be related to self-sized corrugating medium. Furthermore, the sizing manifests itself in more than two-thirds of the cases as an adhesion problem between the fluted medium and linerboard.

Corrugating manufacturers use a variety of remedial measures to cope with medium which becomes self-sized. The most frequent control method involves addition of a rewetting agent to the pulp prior to sheet formation. Machine processing changes (more steam and heat) during the converting operation and reduced roll inventory (less storage time) are also employed. Wood selection, pulping conditions, pulp fractionation (removal of fines), improved washing, and control of inorganic content can be optimized to reduce the tendency for self-sizing.

Economical and ecological pressures have encouraged replacement of the standard neutral sulfite semichemical (NSSC) pulping process with more alkaline pulping conditions such as GLSC and nonsulfur conditions such as AC pulps. Also, more recycled fiber is being used. Practically no published information is available regarding how these pulping changes may affect the extent and rate of self-sizing. This information is essential for efficient control of self-sizing.

A research plan based on the survey information was formulated. The initial part of this research program was concerned with the comparative study of the self-sizing characteristics of representative NSSC, GLSC, AC, and recycled pulps. The effects of stock pH in conjunction with additions of rewetting agents were also determined. The quantity and type of organic solvent extractable material was also determined. Specifically, the project goals are:

TABLE I
SUMMARY OF SURVEY RESULTS

Questionnaire Response

Number of corrugating manufacturers sampled	45
Number of responses	69%

Occurrence of Self-Sizing

	Yes, %	No, %	Don't Know
Currently experiencing problem?	43	39	18
Experienced problem in past?	64	24	12

Problems Related to Self-Sizing

Adhesion problems	67
Poor fluting	25
Other	8

Processing Variables which are Altered
to Accommodate Self-Sized Mediums

Adhesive consumption and formulation changes	35
Increased energy consumption (steam & heat)	32
Reduce machine speed	22
Other	11

Remedial Measures

Use of rewetting agents	58
Controlled pulping	16
Short inventory times	10
Other	16

Effect of Pulp Stock

Correlation of self-sizing with pulp stock	55	24	21
NSSC pulps	33		
Green liquor pulps	33		
Recycled fiber pulps	23		
Other	11		

Solution of Self-Sizing Problem

Should The Institute of Paper Chemistry pursue a program on self-sizing corrugating medium?	60	20	20
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1. To evaluate the self-sizing potential of conventional NSSC,
selected alkaline, and recycled fiber pulps
2. To establish economical procedures for treating the
problem

EXPERIMENTAL

PULP PROCESSING AND ANALYSIS

Based on the mill survey and other contacts with industry representatives, five pulps were selected for study in the self-sizing program. These included a conventional NSSC, two GLSC, one AC, and one recycled fiber pulp.

Most of the pulps selected for study were received in a dewatered condition. The equivalent of five pounds of the dewatered pulp (dry basis) was refined in filtered tap water at approximately 3% consistency to a freeness range of 225-290 mL CSF. A Valley beater was used for this purpose. The beaten pulps were then dewatered to 18-26% consistency, and formaldehyde was added in an amount equivalent to 0.37% based on dry fiber weight. The dewatered pulp was stored at 40°F until used in subsequent analysis and/or handsheet preparations. Fiber analysis of the pulps is recorded in Table II. Chemical analysis of the pulps by emission spectroscopy is presented in Table III.

All pulps were solvent extracted to determine the amount and type of resinous materials which could contribute to self-sizing. In this direction the pulps were first washed with ethanol to remove water, followed by extraction with 2:1 benzene-alcohol in a Soxhlet extractor. The alcohol washings were combined with the benzene-alcohol extracts and the combination was reduced to dryness over steam. The composition of the extracts was determined by the classic separation procedure, and the results are recorded in Table IV.

The acidity or alkalinity of the five refined pulps and handsheets prepared therefrom was determined according to TAPPI Standard Method T 428 pm-77, and the results are presented in Table V.

TABLE II
FIBER ANALYSIS OF PULPS

Pulp	Designation or Abbreviation	Fiber Type, %	Principal	Other
Neutral sulfite semichemical	NSSC	Hardwood, 100	Oak	Beech, red gum, black gum, maple
Green liquor semichemical	GLSC-1 (Source 1)	Hardwood chemi- mechanical, 100	Red gum	Oak, black gum
	GLSC-2 (Source 2)	Hardwood chemi- mechanical, 100	Oak	Yellow poplar, maple, red gum, black gum
Alkali carbonate	AC	Hardwood chemi- mechanical, 100	Populus	Birch, maple
Recycled container board	Recycled	Softwood kraft, 75.5	Hard pine	Douglas-fir
		Hardwood kraft, 11.0	Black gum, red gum	Maple, oak
		Hardwood NSSC, 13.5	Populus, red gum, black gum	Beech, maple

TABLE III
INORGANIC COMPOSITION OF PULP - EMISSION SPECTROSCOPY

Pulp	Ash, %	ppm												
		B	Si	Mn	Al	Fe	Mg	Pb	Cr	V	Ca	Cu	Na	Ti
NSSC	2.0	--	400	140	20	190	140	47	9.3	--	4800	110	2200	--
GLSC-1	1.6	2.5	350	110	200	100	360	1.3	4.8	2.5	5200	37	510	2.4
GLSC-2	1.8	1.6	86	95	31	37	310	31	2.8	--	3700	61	1800	1.6
Alkaline carbonate	0.9	0.62	110	34	6.4	63	220	1.0	4.9	--	1400	48	1000	--
Recycled	2.0	11	2800	64	2300	370	430	67	8.7	--	2000	110	1100	340

TABLE IV
CHEMICAL COMPOSITION OF BENZENE-ETHANOL EXTRACTS OF PULPS

Pulp	Total Extractives, %	Free Acids, %	Neutrals, %	Unsaponifiables	Combined Acids, %	Water-Soluble Portion, %
NSSC	4.69	0.46	0.14	0.083	0.088	4.09
GLSC-1	5.47	0.68	0.16	0.099	0.052	4.63
GLSC-2	9.20	0.89	0.18	0.10	0.096	8.13
Alkali- carbonate	4.44	0.38	0.15	0.084	0.062	3.91
Recycled	2.78	0.45	0.63	0.44	0.12	1.70
Classified ^a NSSC	1.50	0.20	0.10	0.024	0.033	1.20
Classified ^a GLSC-1	3.55	0.36	0.08	0.025	0.050	3.11
Classified ^a GLSC-2	4.49	0.52	0.04	0.040	0.034	3.88
Classified ^a Alkali car- bonate	1.95	0.20	0.08	0.034	0.023	1.67

^aMaterial retained on combination of sieves.

TABLE V

ACIDITY OR ALKALINITY OF PULP AND HANDSHEET EXTRACTS

Pulp Sample	pH of Pulp as Received	SO ₃ , %	Na ₂ O, %
NSSC	6.0	0.0028	
GLSC-1	7.4	--	0.0098
GLSC-2	7.6	--	0.0096
Alkali carbonate	7.4	--	0.0059
Recycled	5.4	0.0112	
Handsheet Sample ^a			
GLSC-1	--	--	0.0211
GLSC-2	--	--	0.0434
Alkali carbonate	--	--	0.0149

^aPrepared at pH 11.

HANDSHEET PREPARATIONS AND TESTING

All handsheets were prepared in a Rapid-Kothen mold (8-inch diameter). The required amount of dewatered pulp for a given series of handsheets (usually 25 grams o.d. basis) was dispersed at 1.25% consistency in a British disintegrator for a total of 300 counts. The pulp was then transferred to a stainless steel container where it was diluted to 0.5% consistency with room-temperature deionized water. The pH of the pulp was adjusted to the desired level, and additives, when employed, were stirred in for a minimum of five minutes before handsheet preparation. Sufficient pulp for a single handsheet was then metered into the sheet mold. Deionized water preadjusted to the same pH level as that of the pulp slurry was added, resulting in a forming consistency of 0.1%. The fiber slurry in the mold was stirred for 10 minutes

with an agitator consisting of four paddles set at an angle of 90 degrees to each other and centered in the sheet mold about 0.25 inch from the 100-mesh stainless steel forming wire. The paddles were power driven and mechanically arranged so as to produce an oscillating motion covering a sweep of 60 degrees. As soon as the agitation was stopped, the stirrer was removed from the mold and the fiber drained immediately onto the forming wire. The handsheet weight was 3.85 g (o.d. basis), equivalent to 134 g/m². After forming, the sheets were couched from the wire onto blotter stock and then pressed for five minutes at 50 lb pressure. The sheets were dried in contact with one blotter on a steam-heated drum for seven minutes at 105°C.

Porosity and smoothness are among those properties known to affect wetting and penetration of liquids in porous media. Accordingly, porosity and smoothness measurements were made on handsheets formed from the selected pulps before and after solvent extraction or fiber classification. Results are recorded in Table VI.

In most cases water absorbency was measured on sheets aged at 23°C and 50% RH and on sheets subjected to accelerated aging.

Accelerated aging of handsheets was carried out in an oven at 105°C. Sheets were cut into strips (1-1/2 by 3 inches) stacked six deep and placed in a clean 100 by 15-mm Petri dish. The Petri dishes were cleaned in chromic acid cleaning solution followed by rinsing with tap water and finally with distilled water. The dishes were then allowed to air dry. The covered dishes containing the handsheet strips were placed in an oven at 105°C. After 30 minutes, the sample under the top strip was removed and the other five were left in the oven for further aging. Three additional strips were removed in the same manner after oven aging 1.0, 2.0, and 4.0 hours. The oven-aged strips were conditioned at 73°F and 50% RH a minimum of 30 minutes before testing for water resistance.

TABLE VI
SELECTED PROPERTIES OF HANDSHEETS PREPARED
FROM VARIOUS PULPS

Pulp Used to Prepare Handsheets	pH	Bendtsen Porosity, mL/min (wire side)	Bendtsen Smoothness, mL/min	
			Wire	Felt
NSSC	7	74	3373+	3373+
NSSC	11	137	3373+	3373+
GLSC-1	7	186	3373+	2983
GLSC-1	11	235	3373+	3086
GLSC-2	7	<18	3373+	3373+
GLSC-2	11	<18	3373+	3310
Alkali-carbonate	7	283	3180	2850
Alkali-carbonate	11	268	3220	2680
Recycled	7	129	2913	2550
Recycled	11	82	3016	2545
Extracted NSSC ^a	7	117	3373+	3373+
Extracted GLSC-1 ^a	7	496	3373+	3046
Extracted GLSC-2 ^a	7	<18	3373+	3373+
Extracted alkali- carbonate ^a	7	236	3268+	2826
Extracted recycled ^a	7	135	2805	2527
Classified NSSC ^b	7	3373+	3373+	3373+
Classified GLSC-1 ^b	11	3373+	3373+	3373+
Classified GLSC-2 ^b	11	3340	3373+	3373+
Classified alkali- carbonate ^b	11	3373+	3271	3201

^aHandsheets were prepared from benzene-alcohol extracted pulps.

^bClassified pulp was that portion retained on combination of sieves.

The degree of water resistance or self-sizing was determined simultaneously by two methods, i.e., through penetration and waterdrop time. For this purpose a 50-microliter drop of room-temperature distilled water was placed on the surface of

the handsheet. Through penetration was determined by measuring the time required for the water to soak through the sheet and appear on the underside. A mirror positioned at an angle under the sheet was used in determining through penetration. The time required for the drop to completely soak into the sheet was recorded as the water-drop time. Emphasis in this report is placed on the waterdrop values.

Sizing values for the NSSC pulp at pH levels of 7, 9, and 11 are presented in Table VII and Fig. 1. Comparable data for the GLSC-1, GLSC-2, alkali-carbonate, and recycled fiber pulps are recorded in Tables VIII-XI and Fig. 2-4. Note: No figure is included for the GLSC-2 pulp, since the waterdrop values were 600+ seconds without aging.

TABLE VII
WATER ABSORBENCY IN HANDSHEETS PREPARED FROM
CONVENTIONAL NSSC PULP

		Aging Temperature											
		23°C						105°C					
pH	Aging Time, hr	Through Penetration, sec			Waterdrop, sec			Through Penetration, sec			Waterdrop, sec		
		Wire	Felt	Av.	Wire	Felt	Av.	Wire	Felt	Av.	Wire	Felt	Av.
7	0.5	4	3	4	169	133	151	21	19	20	600+	466	533+
	1.0	4	3	4	165	157	161	45	47	46	600+	600+	600+
	2.0	3	2	3	141	127	134	600+	458	529+	600+	600+	600+
	4.0	3	3	3	185	156	171	600+	600+	600+	600+	600+	600+
9	0.5	3	2	3	229	147	188	3	2	3	307	236	272
	1.0	3	2	3	273	150	214	7	9	8	580	325	453
	2.0	3	2	3	272	148	220	34	21	28	600+	600+	600+
	4.0	3	2	3	283	160	222	600+	600+	600+	600+	600+	600+
11	0.5	4	3	4	200	179	190	10	11	11	163	176	170
	1.0	3	3	3	200	172	186	15	11	13	242	198	219
	2.0	3	2	3	211	145	178	18	13	16	405	287	346
	4.0	3	2	3	188	155	172	17	14	16	513	369	441

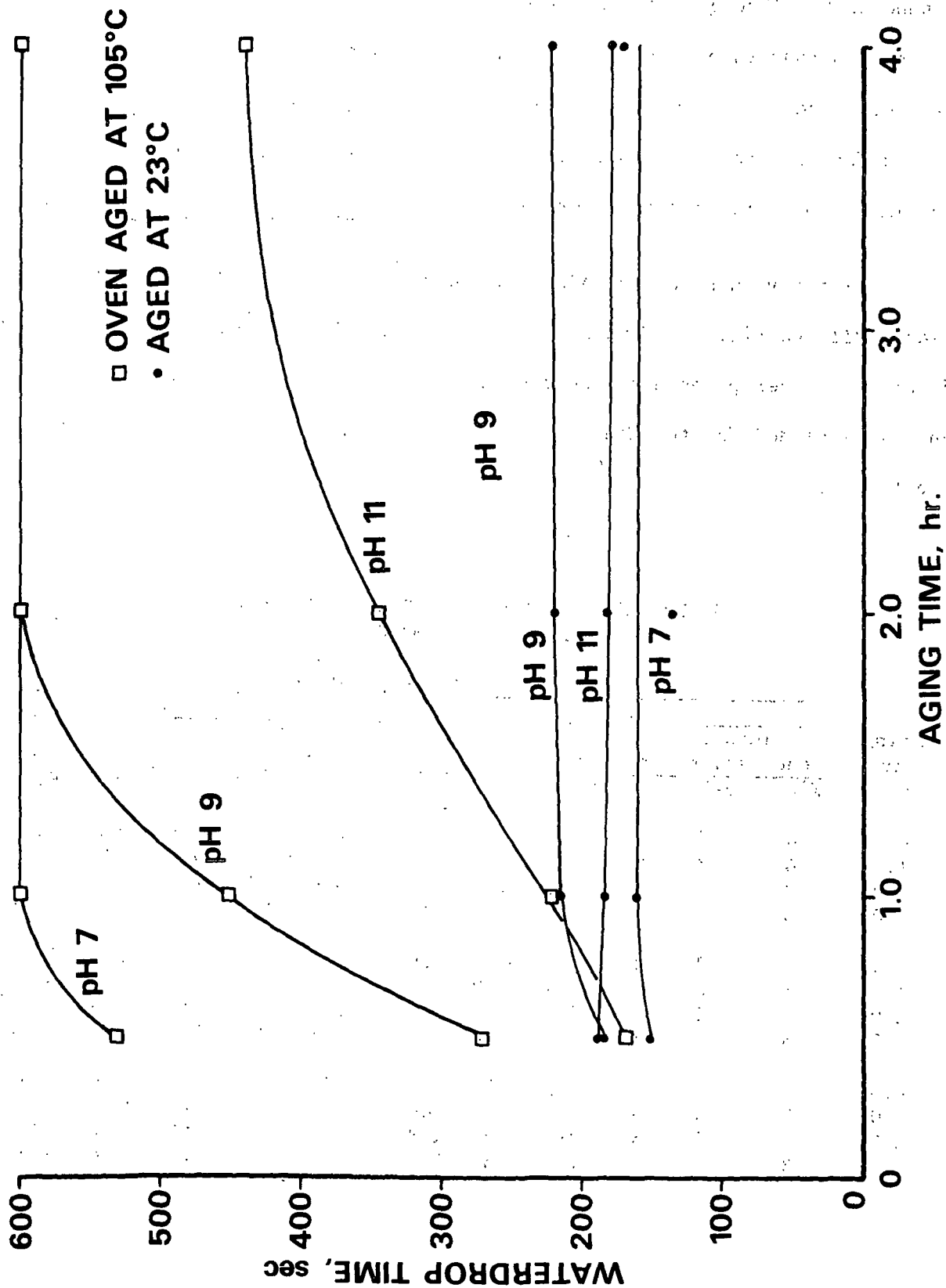


Figure 1. The effect of aging on waterdrop absorbency of handsheets prepared from NSSC pulp.

WATER ABSORBENCY IN HANDSHEETS PREPARED FROM GREEN LIQUOR SEMICHEMICAL PULP (GLSC-1)

[illegible]

WATER ABSORBENCY IN HANDSHEETS PREPARED FROM GREEN LIQUOR SEMICHEMICAL PULP (GLSC-2)

[illegible]

TABLE X

WATER ABSORBENCY IN HANDSHEETS PREPARED FROM
ALKALI-CARBONATE PULP

		Aging Temperature											
		23°C						105°C					
pH	Aging Time, hr	Through Penetration, sec			Waterdrop, sec			Through Penetration, sec			Waterdrop, sec		
		Wire	Felt	Av.	Wire	Felt	Av.	Wire	Felt	Av.	Wire	Felt	Av.
7	0.5	2	2	2	39	34	27	3	2	3	80	39	60
	1.0	2	2	2	35	33	34	11	9	10	198	163	181
	2.0	2	2	2	45	31	38	28	30	29	600+	600+	600+
	4.0	2	2	2	49	48	49	135	72	104	600+	600+	600+
9	0.5	2	2	2	51	38	45	2	2	2	109	76	93
	1.0	2	2	2	52	36	44	8	8	8	248	160	204
	2.0	2	2	2	65	41	53	28	29	29	600+	600+	600+
	4.0	2	2	2	55	45	50	58	66	62	600+	600+	600+
11	0.5	2	2	2	93	70	82	5	5	5	249	167	208
	1.0	2	2	2	97	80	89	10	10	10	381	304	343
	2.0	2	2	2	121	90	106	21	18	20	600+	600+	600+
	4.0	2	2	2	103	105	104	41	47	44	600+	600+	600+

TABLE XI

WATER ABSORBENCY IN HANDSHEETS PREPARED FROM
RECYCLED FIBER PULP

		Aging Temperature											
		23°C						105°C					
pH	Aging Time, hr	Through Penetration, sec			Waterdrop, sec			Through Penetration, sec			Waterdrop, sec		
		Wire	Felt	Av.	Wire	Felt	Av.	Wire	Felt	Av.	Wire	Felt	Av.
7	0.5	1	1	1	87	35	61	1	1	1	87	41	64
	1.0	1	1	1	98	49	74	1	1	1	103	47	75
	2.0	1	1	1	107	51	79	1	1	1	108	46	77
	4.0	1	1	1	89	51	70	1	1	1	117	57	87
9	0.5	1	1	1	89	44	67	1	1	1	107	55	81
	1.0	1	1	1	94	33	64	1	1	1	89	51	70
	2.0	1	1	1	85	37	61	1	1	1	90	53	72
	4.0	1	1	1	85	42	64	1	1	1	115	68	92
11	0.5	1	1	1	59	34	47	1	1	1	85	50	68
	1.0	1	1	1	42	33	38	1	1	1	92	49	71
	2.0	1	1	1	54	31	43	1	1	1	94	56	75
	4.0	1	1	1	60	31	46	1	1	1	102	53	78

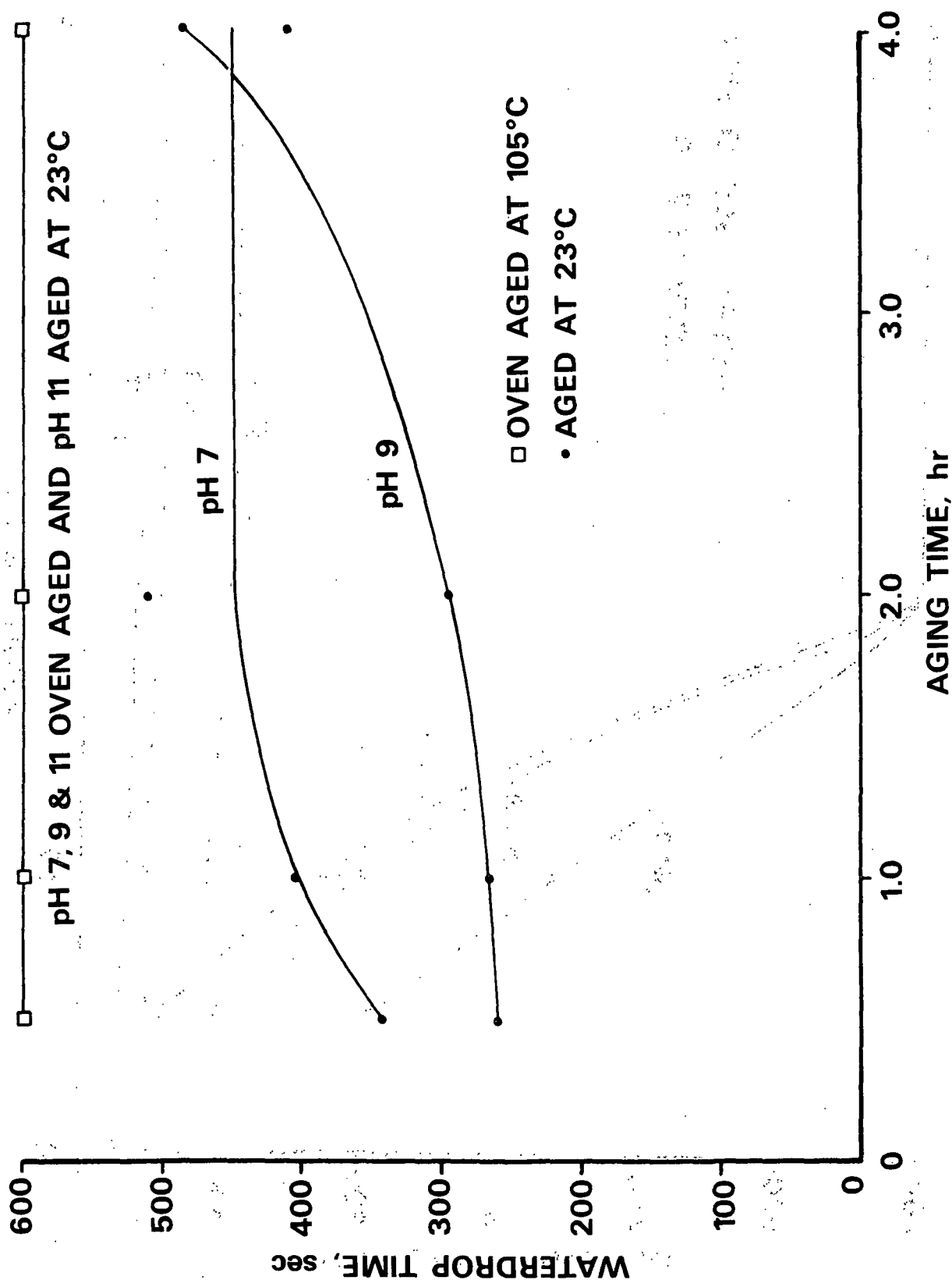


Figure 2. The effect of aging on waterdrop absorbency of handsheets prepared from GLSC-1 pulp.

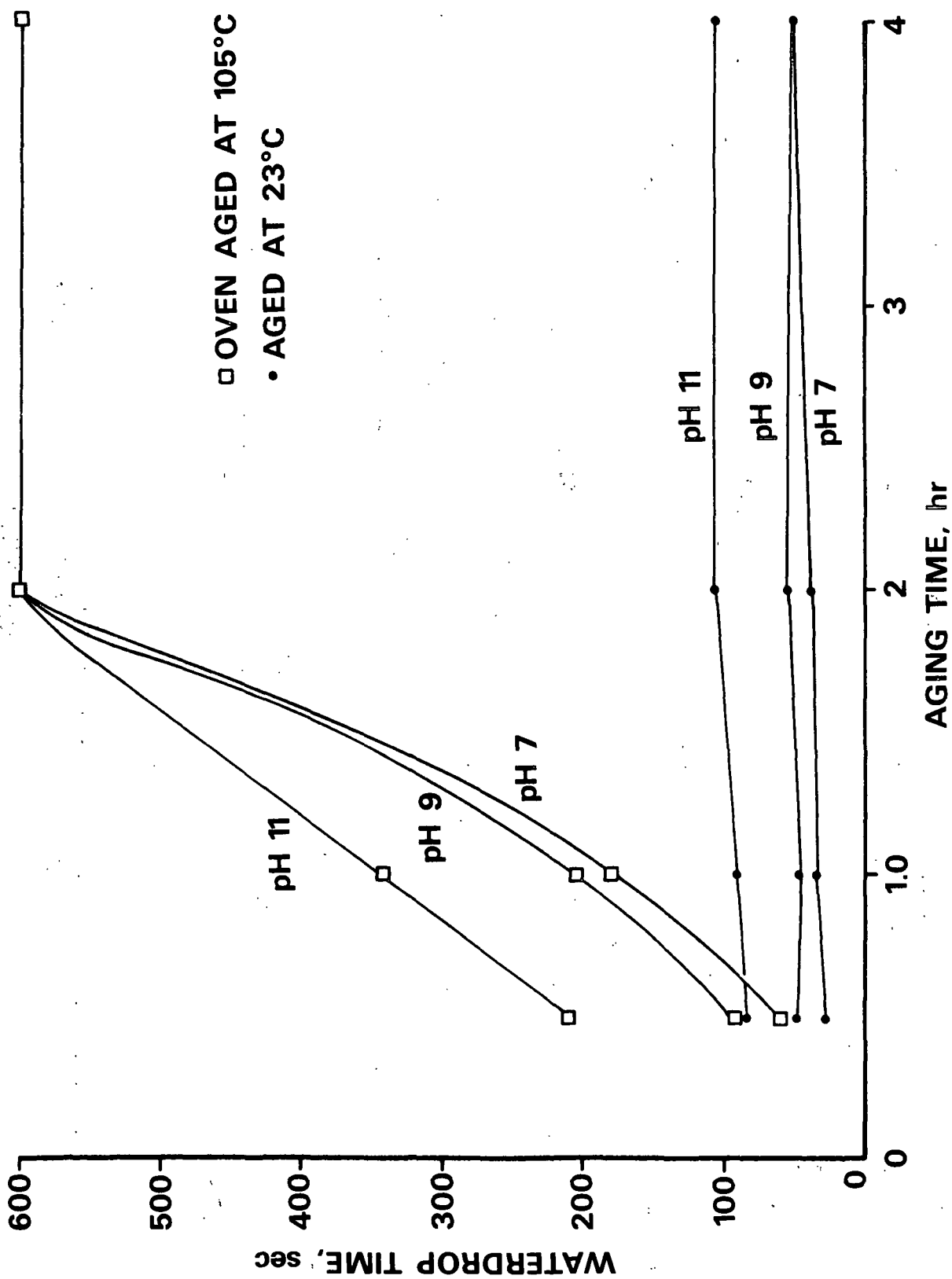


Figure 3. The effect of aging on waterdrop absorbency of handsheets prepared from alkali-carbonate pulp.

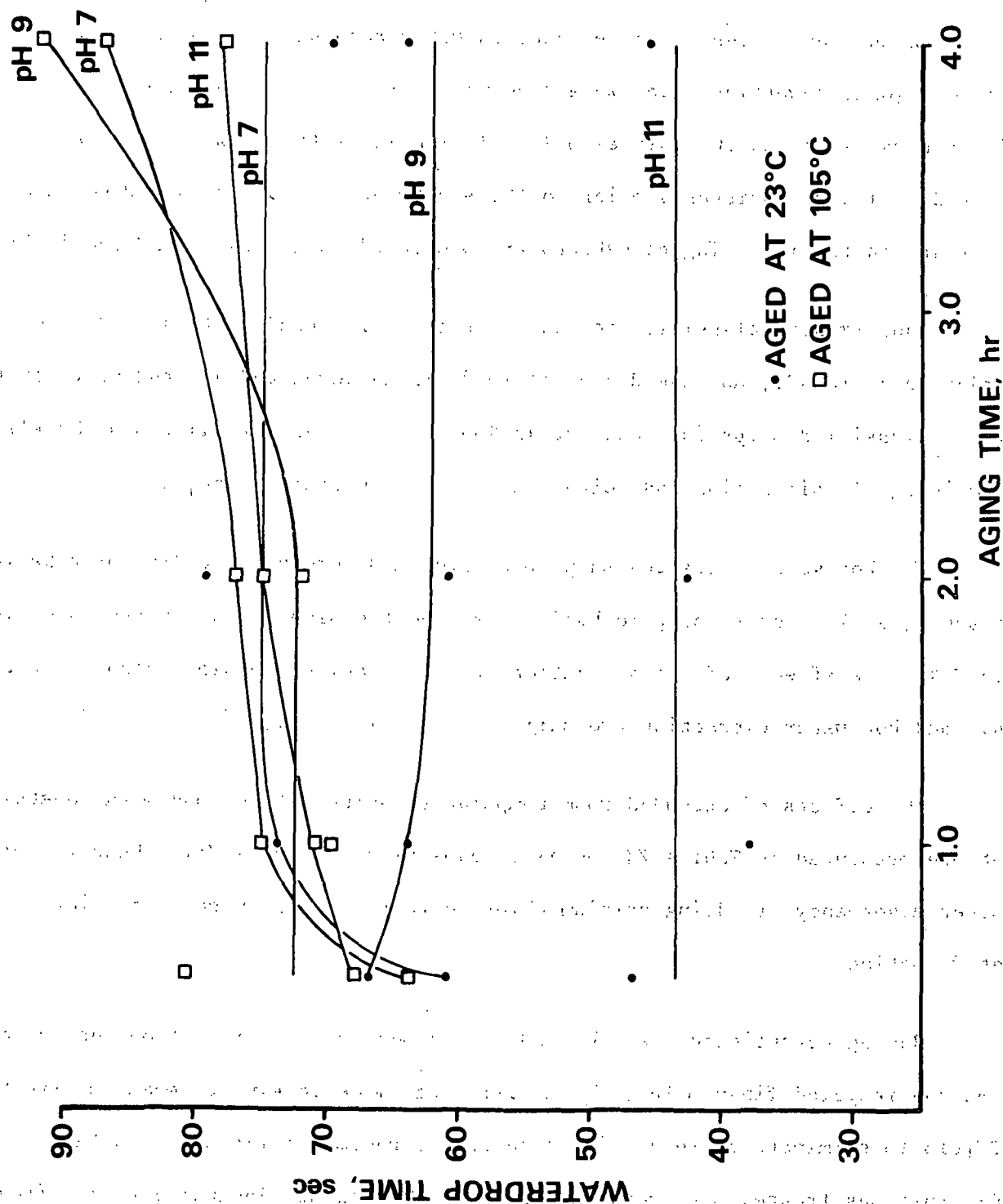


Figure 4. The effect of aging on waterdrop absorbency of handsheets prepared from recycled fiber pulp.

In subsequent tests, the effects on water absorbency of a commercially accepted anionic rewetting agent were examined over a range in addition level. The surfactant was added to the pulp as a 1% solution in distilled water. Addition was made at 0.5% fiber consistency prior to the sheet-forming operation. Water absorbency tests on the resulting handsheets are presented in Tables XII-XV and in Fig. 5-7.

Bauer-McNett classification data for the pulps used in this study are recorded in Table XVI, and the degree of self-sizing developed in handsheets prepared from the classified pulps is presented in Table XVII. Table XVIII shows the effect of combining classification and solvent extraction on sheet absorbency.

The hot water-extracted pulps produced in determining acidity or alkalinity were subsequently used to prepare handsheets, and the size tests are presented in Table XIX. The effects of classification, classification + benzene-alcohol extraction (B/A), and hot water extraction are summarized in Fig. 8-11.

The effects of extended room-temperature aging without and with rewetting agent are presented in Tables XX and XXI, respectively. Table XXII shows the effect on water absorbency of adding calcium chloride to the fiber suspension prior to sheet formation.

Having established that the GLSC pulps were notably more troublesome than the AC and recycled fiber pulps, the decision was made to explore means of treating GLSC pulp to eliminate or reduce its tendency to become self-sized. In this direction, GLSC-1 pulp was treated with acid and several other agents including Calgon (sodium hexametaphosphate), Versene (tetra-sodium salt of ethylenediaminetetracetic acid), and mixtures of Calgon and the anionic rewetting agent. The effect of pulp temperature at the time of sheet formation was also examined. For purposes of comparison, reference is made to the aging results for the untreated pulp previously presented in Table VIII.

TABLE XII
WATER ABSORBENCY IN HANDSHEETS PREPARED FROM NSSC PULP
INCORPORATING AN ANIONIC RETWETTING AGENT AT PH 7

Rewetting Agent, %	Aging Time, hr	Aging Temperature									
		23°C					105°C				
		Through Penetration, sec		Waterdrop, sec		Wire	Through Penetration, sec		Waterdrop, sec		Wire
		Felt	Av.	Felt	Av.		Felt	Av.	Felt	Av.	
0.0	0.5	4	3	4	169	133	21	19	20	466	533
	1.0	4	3	4	165	157	45	47	46	600+	600+
	2.0	3	2	3	141	127	600+	458	525+	600+	600+
	4.0	3	3	3	185	156	600+	600+	600+	600+	600+
0.025	0.5	2	2	2	212	138	2	2	2	209	269
	1.0	2	1	2	226	150	4	4	4	200	264
	2.0	2	1	2	286	138	30	17	24	600+	600+
	4.0	2	1	2	248	139	600+	600+	600+	600+	600+
0.050	0.5	1	1	1	191	67	1	1	1	126	144
	1.0	--	--	--	--	--	1	1	1	108	125
	2.0	--	--	--	--	--	2	2	2	131	175
	4.0	1	1	1	170	79	180	280	230	600+	600+
0.125	0.5	2	1	2	171	74	2	1	2	77	108
	1.0	--	--	--	--	--	2	1	2	70	101
	2.0	--	--	--	--	--	1	1	1	109	131
	4.0	2	1	2	169	69	7	8	8	406	399

TABLE XIII
WATER ABSORBENCY IN HANDSHEETS PREPARED FROM NSSC PULP
INCORPORATING AN ANIONIC RETWETTING AGENT AT pH 11

Rewetting Agent, %	Aging Time, hr	Aging Temperature											
		23°C						105°C					
		Through			Waterdrop, sec.			Through			Waterdrop, sec.		
		Penetration, sec	Wire	Felt	Av.	Penetration, sec.	Wire	Felt	Av.	Penetration, sec.	Wire	Felt	Av.
0.0	0.5	4	3	4	200	179	190	10	11	11	163	176	170
	1.0	3	3	3	200	172	186	15	11	13	242	198	219
	2.0	3	2	3	211	145	178	18	13	16	405	287	346
	4.0	3	2	3	188	155	172	17	14	16	513	369	441
0.025	0.5	1	1	1	99	48	74	2	2	2	125	110	118
	1.0	1	1	1	107	47	77	10	5	8	372	177	275
	2.0	1	1	1	89	53	71	29	14	22	600+	600+	600+
	4.0	1	1	1	108	55	82	600+	206	403+	600+	600+	600+
0.050	0.5	1	1	1	125	49	88	3	3	3	131	80	106
	1.0	1	1	1	86	52	69	5	4	5	168	128	148
	2.0	2	1	2	124	72	98	11	9	10	428	382	405
	4.0	2	2	2	109	69	89	122	93	108	600+	600+	600+
0.125	0.5	1	1	1	63	34	49	2	2	2	77	71	74
	1.0	1	1	1	53	37	45	2	2	2	127	94	111
	2.0	1	1	1	51	49	50	5	6	6	244	197	221
	4.0	1	1	1	77	49	63	27	25	26	600+	600+	600+

TABLE XIV
WATER ABSORBENCY IN HANDSHEETS PREPARED FROM GLSC-1 PULP
INCORPORATING AN ANIONIC RETWETTING AGENT AT pH 11

Rewetting Agent, %	Aging Time, hr.	Aging Temperature									
		23°C					105°C				
		Through Penetration, sec		Waterdrop, sec		Wire	Through Penetration, sec		Waterdrop, sec		Wire
		Felt	Av.	Felt	Av.		Felt	Av.	Felt	Av.	
0.0	0.5	18	19	20	600+	600+	600+	207	404+	600+	600+
	1.0	22	23	24	600+	600+	600+	247	424+	600+	600+
	2.0	24	23	22	600+	600+	600+	600+	600+	600+	600+
	4.0	26	25	23	600+	600+	600+	600+	600+	600+	600+
0.05	0.5	2	2	2	203	183	193	2	2	272	170
	1.0	3	3	3	473	337	405	10	5	547	287
	2.0	3	3	3	437	305	371	15	10	600+	600+
	4.0	3	3	3	597+	399	498+	17	12	600+	600+
0.075	0.5	1	1	1	288	296	292	2	3	346	271
	1.0	1	1	1	277	272	275	3	3	420	375
	2.0	1	1	1	303	202	253	3	3	417	314
	4.0	1	1	1	271	214	243	6	4	561+	384
0.10	0.5	1	1	1	100	70	85	2	2	128	139
	1.0	1	1	1	115	79	97	2	2	200	169
	2.0	1	1	1	100	50	75	2	2	136	158
	4.0	1	1	1	113	61	87	2	2	216	220
0.125	0.5	1	1	1	39	32	36	1	2	59	54
	1.0	1	1	1	46	35	41	1	2	59	49
	2.0	1	1	1	32	38	35	1	2	59	63
	4.0	1	1	1	36	28	32	1	2	56	64

TABLE XV

WATER ABSORBENCY IN HANDSHEETS PREPARED FROM ALKALI-CARBONATE AND
GLSC-2 PULPS INCORPORATING AN ANIONIC REWETTING AGENT

Pulp Used	Rewetting Agent, %	pH	Through Penetration, sec			Waterdrop, sec		
			Wire	Felt	Av.	Wire	Felt	Av.
Alkali-carbonate	0.0	11	2	2	2	93	70	82
	0.025	11	1	1	1	33	22	28
	0.05	11	1	1	1	29	21	25
	0.125	11	1	1	1	32	22	27
GLSC-2	0.0	7	20	17	19	600+	600+	600+
	0.025	7	12	12	12	600+	600+	600+
	0.05	7	8	8	8	600+	600+	600+
	0.125	7	6	5	6	600+	600+	600+
GLSC-2	0.0	11	600+	600+	600+	600+	600+	600+
	0.025	11	65	55	60	600+	600+	600+
	0.05	11	18	15	17	600+	600+	600+
	0.125	11	8	6	7	565	570	568
	0.25	11	3	3	3	205	200	203

NOTE: All samples aged 1/2 hour at room temperature.

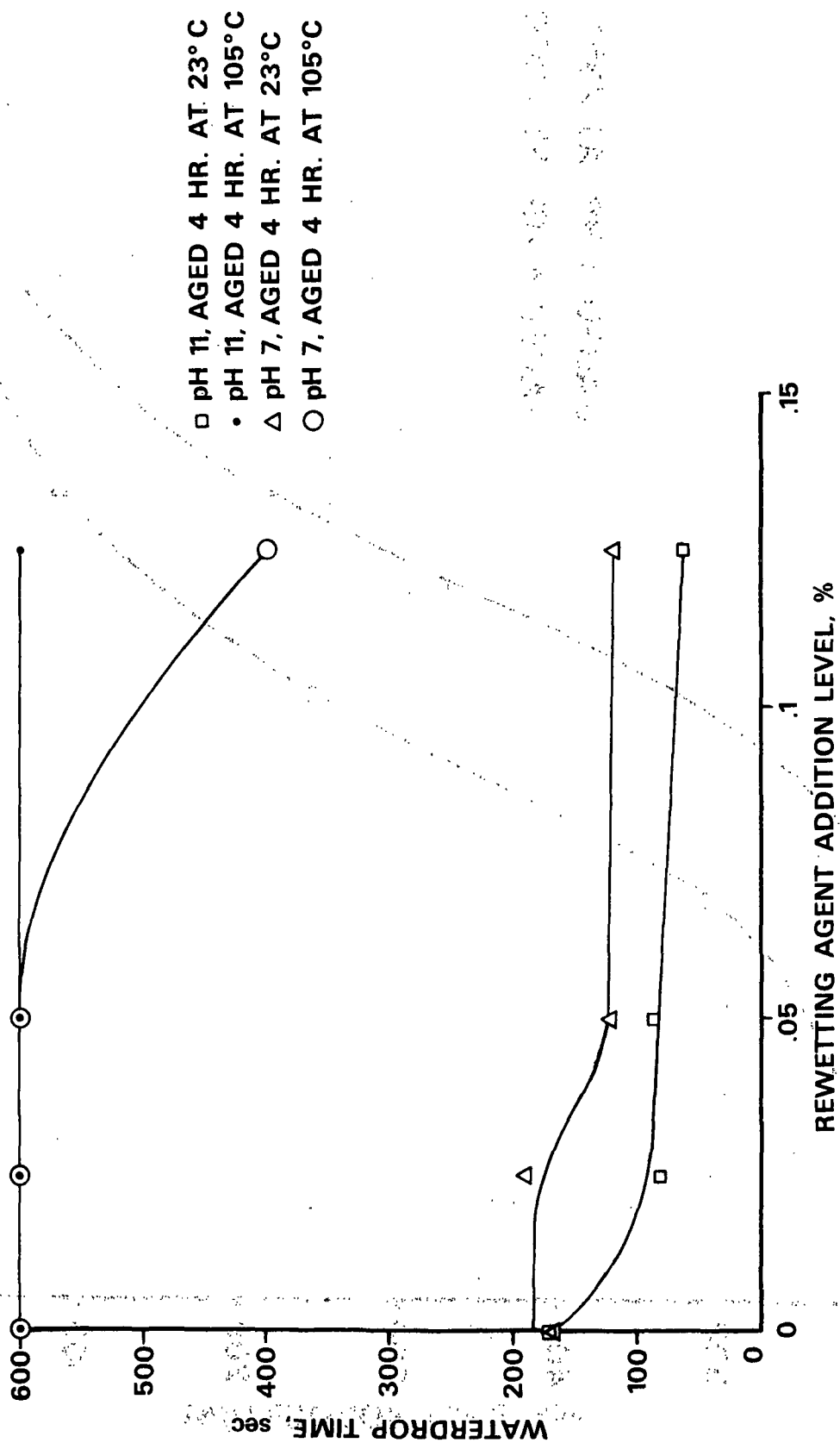


Figure 5. Effect of rewetting agent on waterdrop absorbency of handsheets prepared from NSSC pulp.

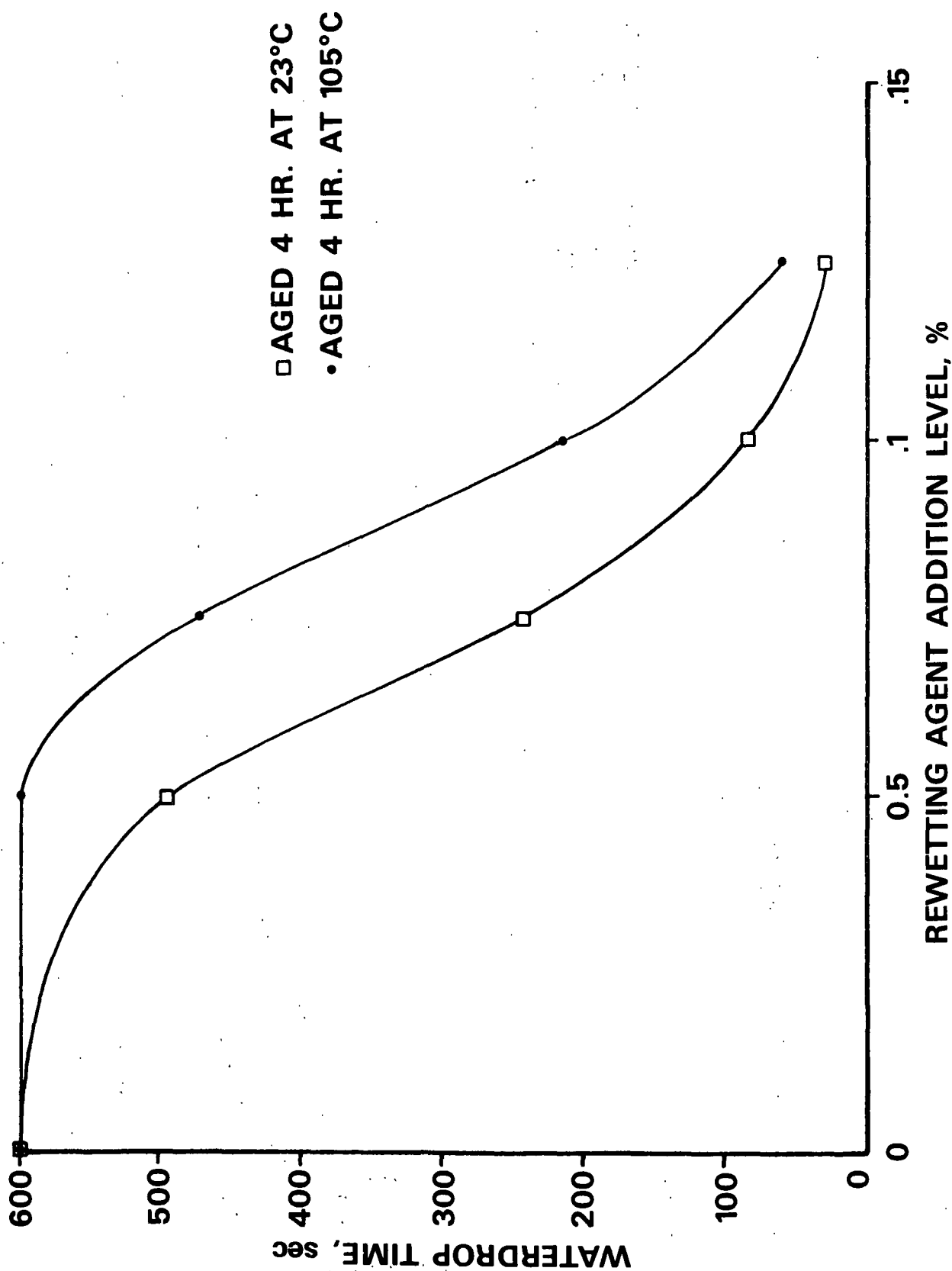


Figure 6. Effect of rewetting agent on waterdrop absorbency of handsheets prepared from GLSC-1 pulp at pH 11.

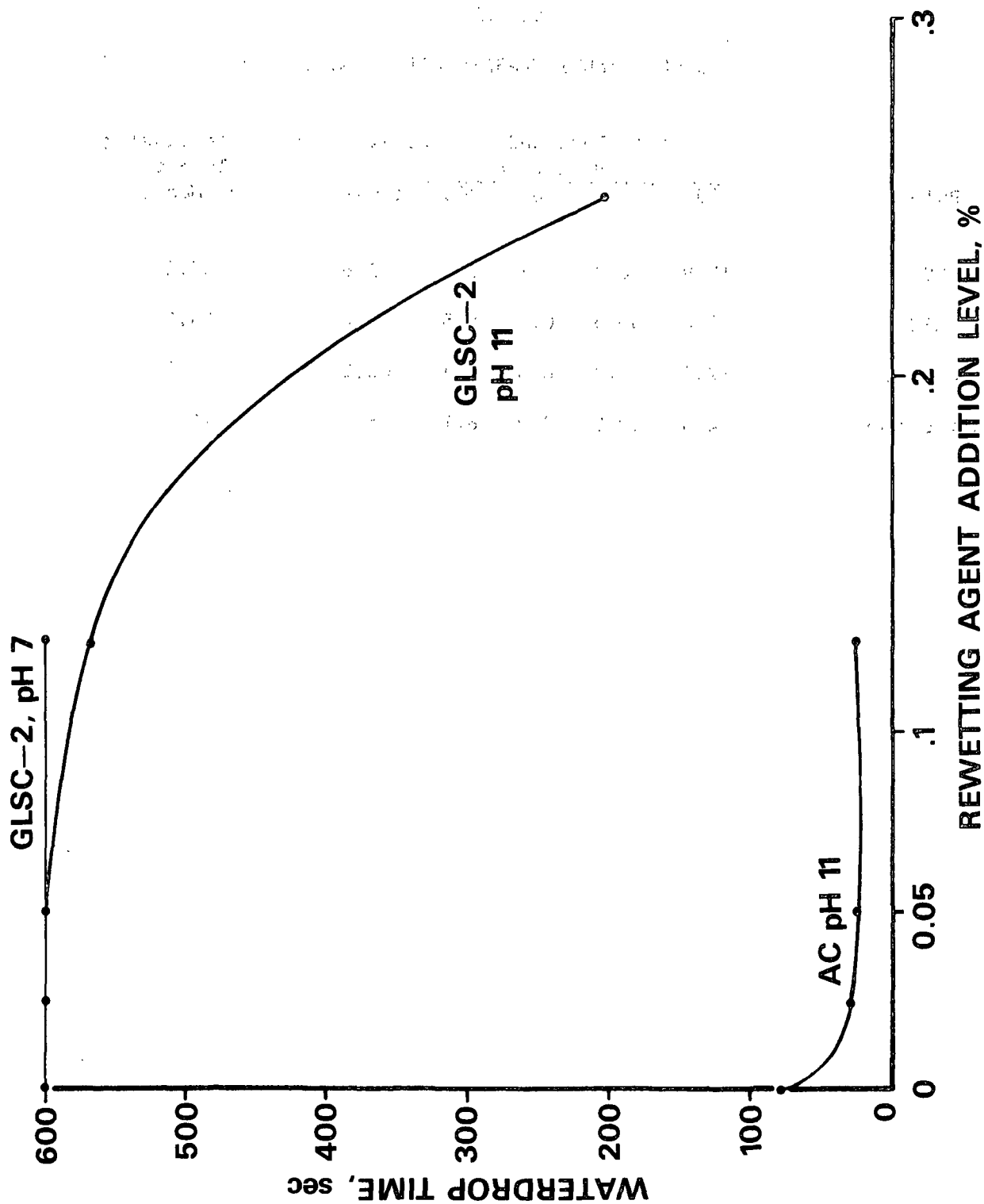


Figure 7. Effects of wetting agent on waterdrop absorbency of handsheets prepared from alkali-carbonate and GLSC-2 pulps.

TABLE XVI
BAUER-McNETT CLASSIFICATION DATA

Pulp	% Pulp Retained on Screens					Pulp Passing Through 200 Mesh, %
	Mesh Size					
	20	48	100	200	Total	
NSSC	12.9	50.7	6.1	3.1	72.8	27.2
GLSC-1	7.0	50.6	10.3	5.5	73.4	26.6
GLSC-2	12.7	41.2	8.0	4.3	66.2	33.8
Alkali-carbonate	4.3	50.0	12.9	8.1	75.3	24.7

TABLE XVII
WATER ABSORBENCY IN HANDSHEETS PREPARED FROM CLASSIFIED PULP

Pulp Used	pH	Aging Time, hr	Aging Temperature												
			23° C						105° C						
			Through			Through			Through			Through			
			Penetration, sec	Waterdrop, sec	Penetration, sec	Waterdrop, sec	Penetration, sec	Waterdrop, sec	Wire Felt	Av.	Wire Felt	Av.	Wire Felt	Av.	
NSSC	7	0.5	--	--	--	--	--	--	--	1	1	1	14	14	14
		1.0	--	--	--	--	--	--	--	1	1	1	19	17	18
		2.0	--	--	--	--	--	--	--	2	3	3	65	58	62
		4.0	--	--	--	--	--	--	--	6	9	8	290	172	231
		24	1	1	1	5	5	5	--	--	--	--	--	--	--
GLSC-1	11	0.5	--	--	--	--	--	--	--	20	10	15	185	392+	289+
		1.0	--	--	--	--	--	--	--	23	20	22	444+	493+	469+
		2.0	--	--	--	--	--	--	--	42	40	41	600+	600+	600+
		4.0	--	--	--	--	--	--	--	75	42	59	600+	600+	600+
		24	20	11	16	313	278	296	--	--	--	--	--	--	--
GLSC-2	11	24	600+	600+	600+	600+	600+	600+	600+	600+	600+	600+	600+	600+	600+
Alkali- carbonate	11	0.5	--	--	--	--	--	--	--	1	1	1	13	10	12
		1.0	--	--	--	--	--	--	--	1	1	1	17	12	15
		2.0	--	--	--	--	--	--	--	1	1	1	20	18	19
		4.0	--	--	--	--	--	--	--	2	2	2	34	28	31
		24	1	1	1	12	10	11	--	--	--	--	--	--	--

TABLE XVIII
WATER ABSORBENCY HANDSHEETS PREPARED FROM CLASSIFIED
AND SOLVENT EXTRACTED PULPS

Pulp Used	Aging Time, hr	Aging Temperature									
		23°C					105°C				
		Through					Through				
		Penetration, sec		Waterdrop, sec		Wire	Penetration, sec		Waterdrop, sec		Wire
		Felt	Av.	Felt	Av.		Felt	Av.	Felt	Av.	
NSSC	0.5	--	--	--	--	--	1	1	1	1	3
	1.0	--	--	--	--	--	1	1	1	1	3
	2.0	--	--	--	--	--	1	1	1	1	4
	4.0	1	1	3	3	3	1	1	1	1	4
GLSC-1	0.5	--	--	--	--	--	1	1	1	1	4
	1.0	--	--	--	--	--	1	1	1	1	3
	2.0	--	--	--	--	--	1	1	1	1	3
	4.0	1	1	3	3	3	1	1	1	1	4
GLSC-2	0.5	--	--	--	--	--	1	1	1	1	4
	1.0	--	--	--	--	--	1	1	1	1	4
	2.0	--	--	--	--	--	1	1	1	1	4
	4.0	1	1	4	3	4	1	1	1	1	5
Alkali- carbonate	0.5	--	--	--	--	--	1	1	1	1	3
	1.0	--	--	--	--	--	1	1	1	1	3
	2.0	--	--	--	--	--	1	1	1	1	3
	4.0	1	1	3	3	3	1	1	1	1	4

TABLE XIX
WATER ABSORBENCY HANDSHEETS PREPARED FROM HOT WATER
EXTRACTED PULPS

Pulp Used	Aging Time, hr	Aging Temperature									
		23° C					105° C				
		Through		Waterdrop, sec		Penetration, sec	Through		Waterdrop, sec		Wire Felt Av.
		Wire	Felt	Av.	Wire		Wire	Felt	Av.	Wire	Felt
NSSC	0.5	--	--	--	--	--	4	6	5	162	138
	1.0	--	--	--	--	--	5	7	6	214	140
	2.0	--	--	--	--	--	11	9	10	258	227
	4.0	5	4	5	162	139	12	10	11	600+	496
GLSC-1	0.5	--	--	--	--	--	1	1	1	57	41
	1.0	--	--	--	--	--	1	1	1	57	52
	2.0	--	--	--	--	--	1	1	1	66	47
	4.0	2	2	2	79	74	1	1	1	69	56
GLSC-2	0.5	--	--	--	--	--	6	5	6	501	207
	1.0	--	--	--	--	--	6	6	6	600+	273
	2.0	--	--	--	--	--	7	9	8	600+	437
	4.0	6	5	6	400	339	12	15	14	600+	600+
Alkali- carbonate	0.5	--	--	--	--	--	1	2	2	54	45
	1.0	--	--	--	--	--	1	1	1	52	43
	2.0	--	--	--	--	--	2	1	2	62	54
	4.0	2	2	2	40	38	3	3	3	98	78
Recycled	0.5	--	--	--	--	--	1	1	1	45	20
	1.0	--	--	--	--	--	1	1	1	40	16
	2.0	--	--	--	--	--	1	1	1	28	18
	4.0	1	1	1	31	15	1	1	1	32	19

NOTE: Handsheets from the NSSC pulp were prepared at pH 7; all others at pH 11.

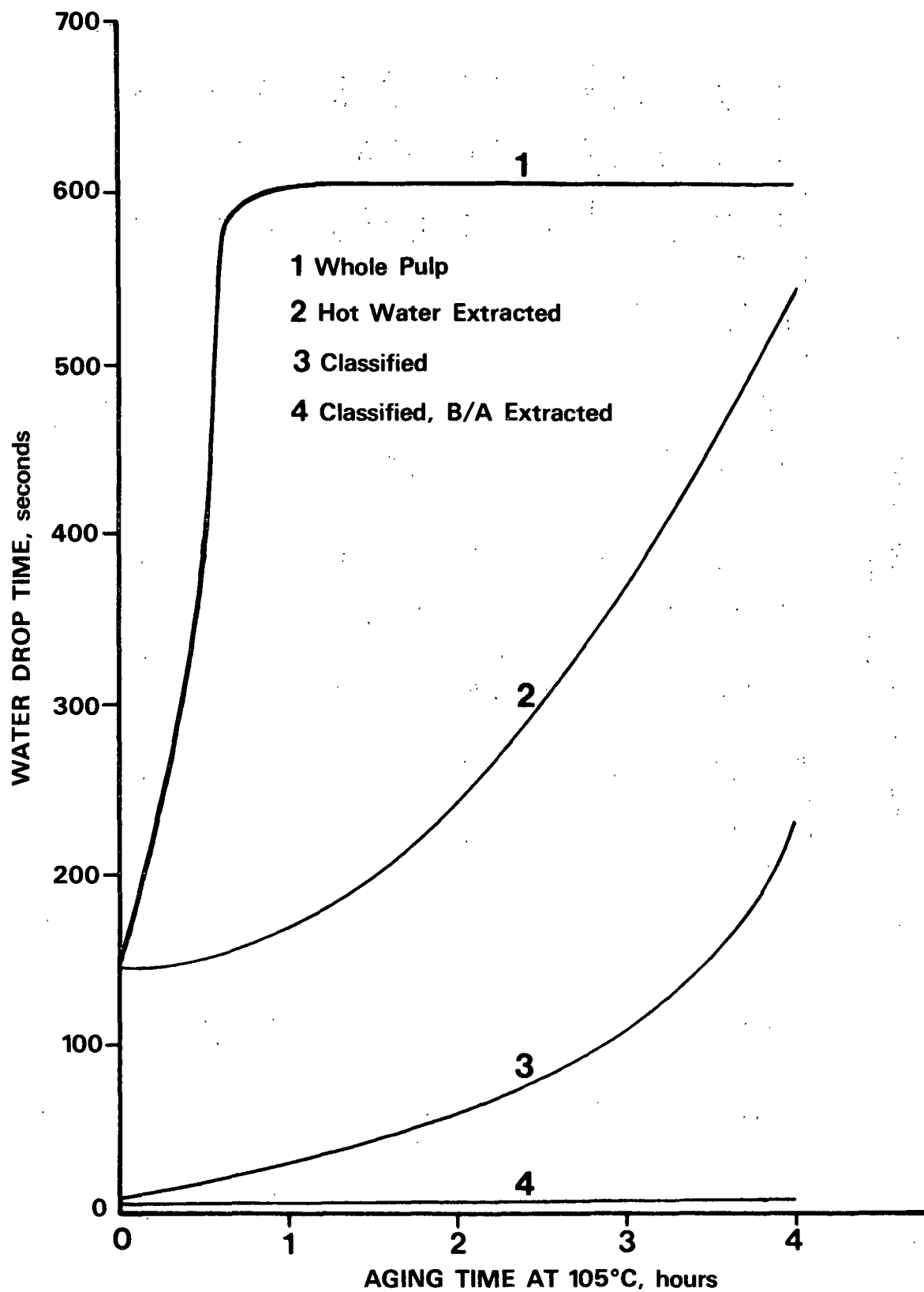


Figure 8. The effect of various treatments on the waterdrop absorbency of NSSC handsheets - pH 7.

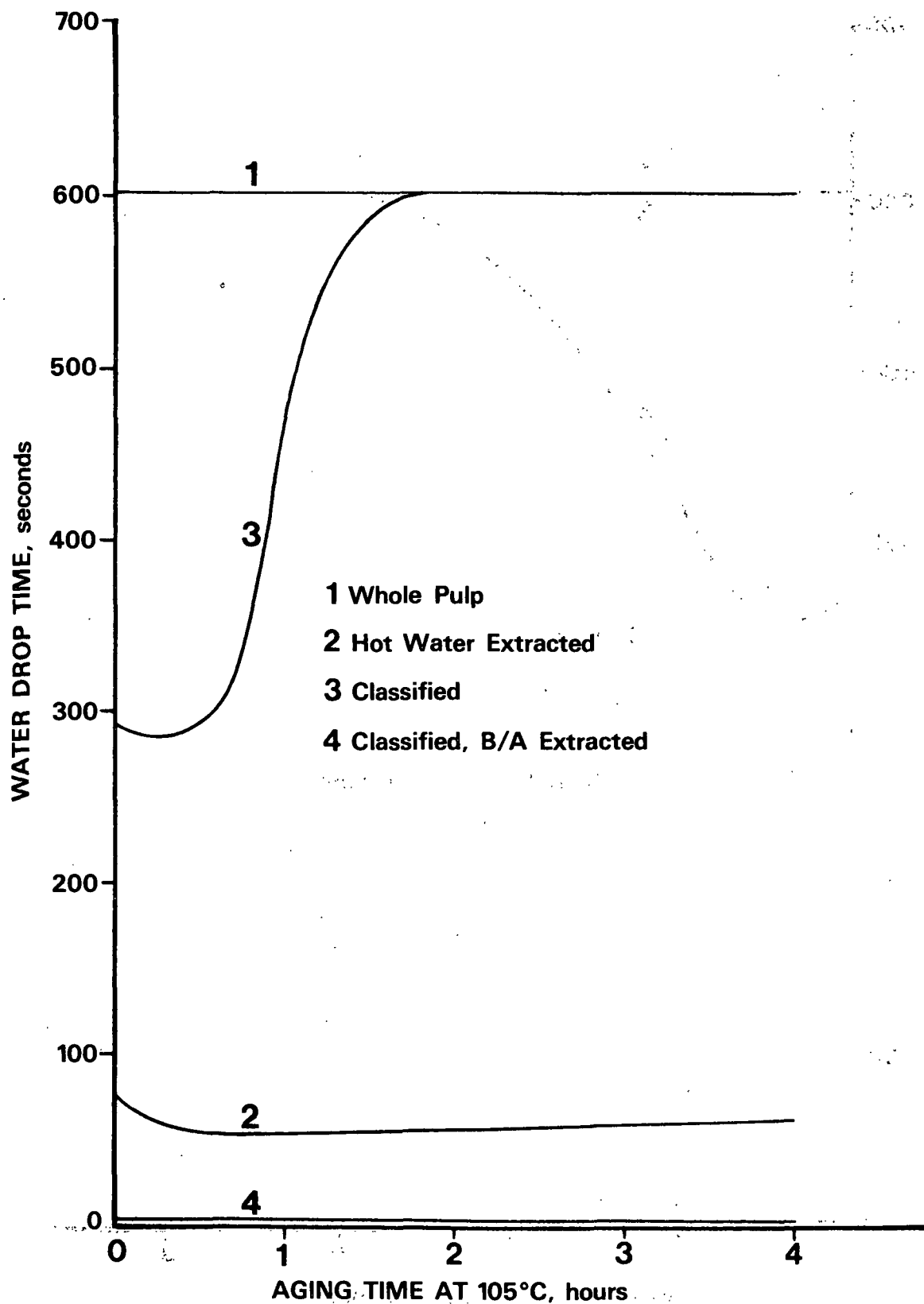


Figure 9. The effect of various treatments on the waterdrop absorbency of GLSC-1 handsheets - pH 11.

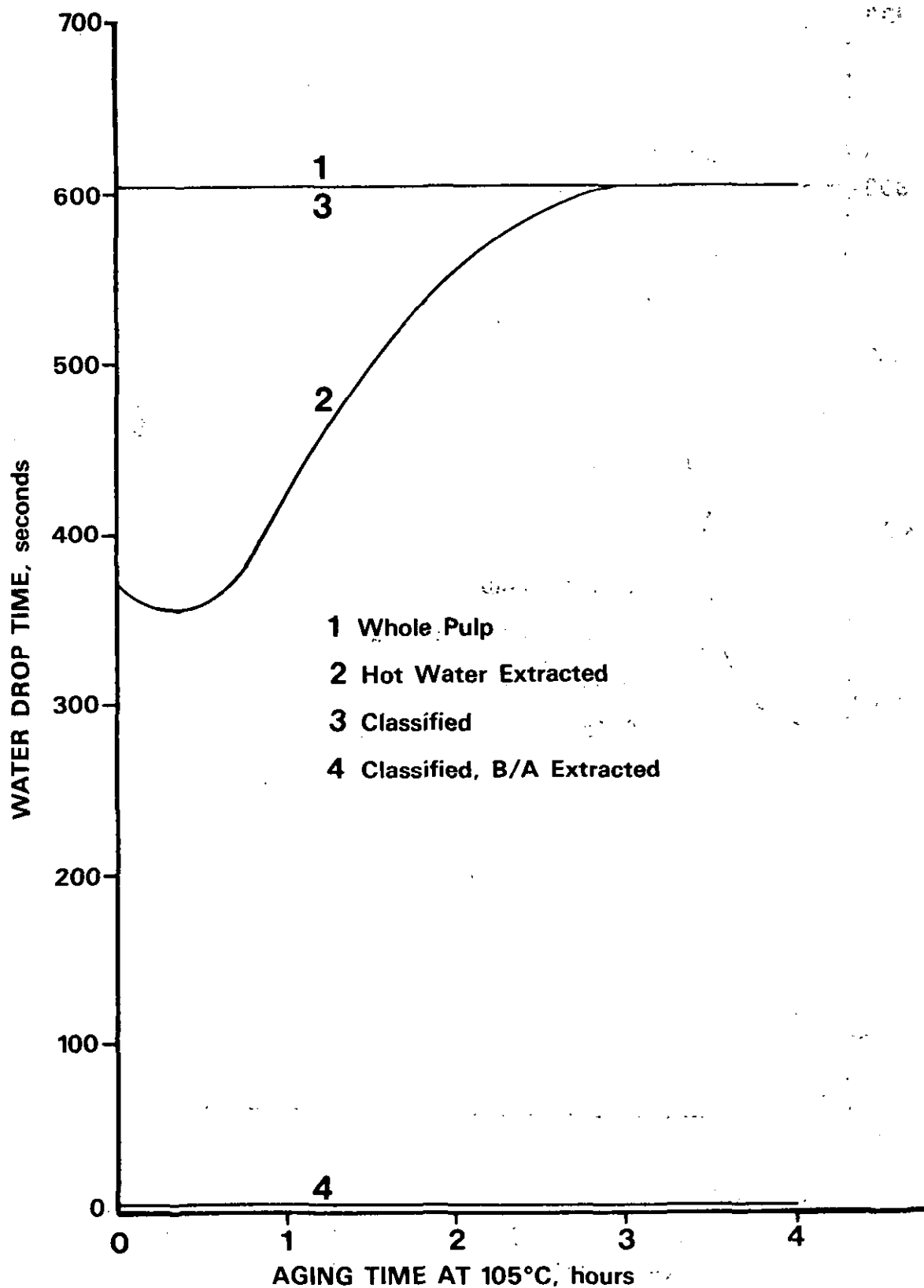


Figure 10. The effect of various treatments on the waterdrop absorbency of GLSC-2 handsheets - pH 11.

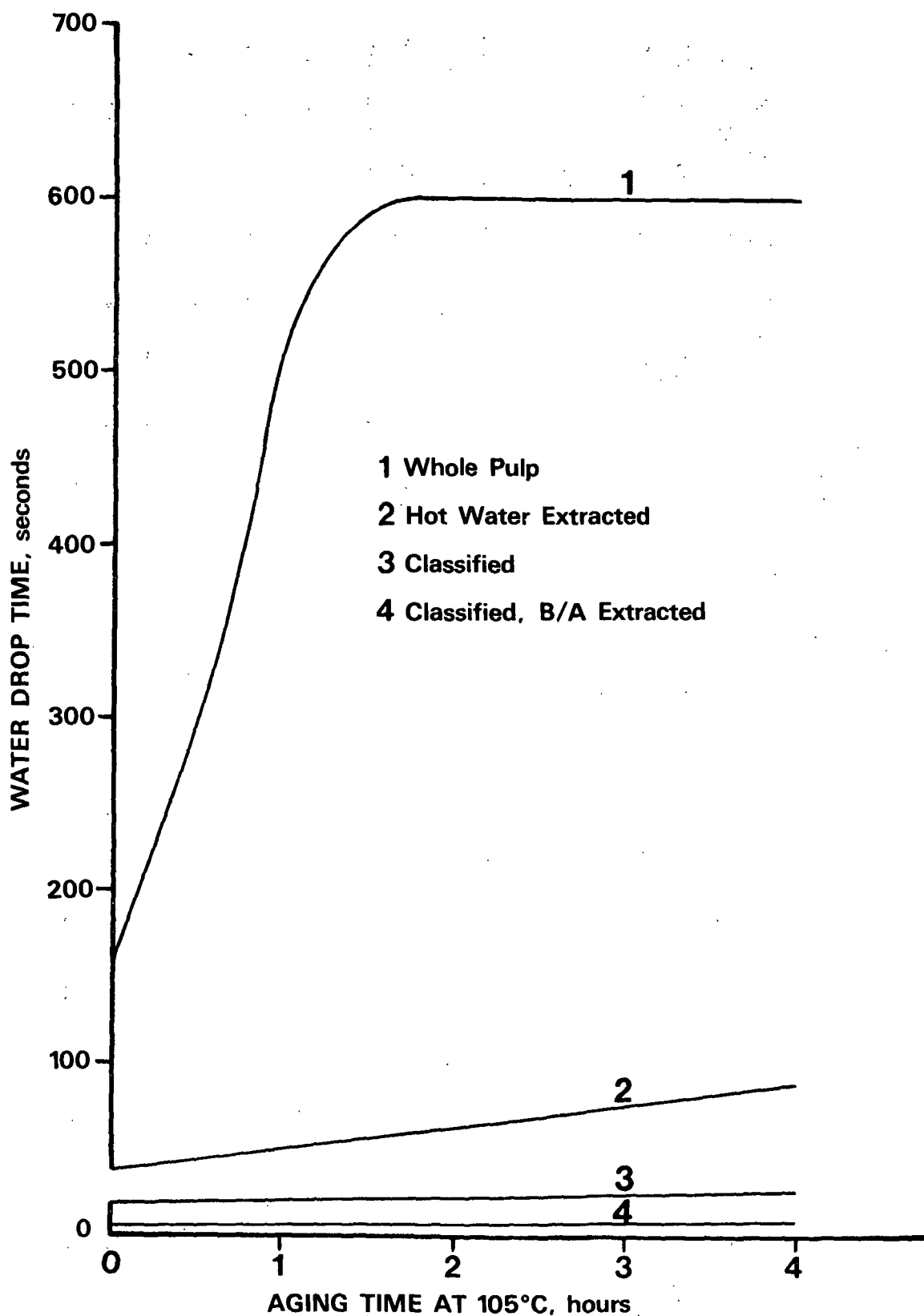


Figure 11. The effect of various treatments on the waterdrop absorbency of AC handsheets - pH 11.

TABLE XX

Pulp Used	pH	Aging Time																	
		4 Hours		2 Weeks		4 Weeks		8 Weeks		12 Weeks		16 Weeks							
		Wire	Felt	Av.	Wire	Felt	Av.	Wire	Felt	Av.	Wire	Felt	Av.	Wire	Felt	Av.			
Waterdrop, sec																			
NSSC	7	185	156	171	313	600+	457	600+	320	460+	160	228	194	184	143	164	316	229	273
NSSC	9	283	160	222	600+	580+	590+	600+	600+	600+	600+	600+	600+	600+	600+	600+	600+	600+	600+
NSSC	11	188	155	172	131	245	188	123	186	155	108	132	120	165	80	123	170	142	156
GLSC-1	7	340	479	410	600+	600+	600+	600+	600+	600+	600+	600+	600+	600+	600+	600+	600+	600+	600+
GLSC-1	9	600+	372	486	600+	600+	600+	600+	600+	600+	600+	600+	600+	600+	600+	600+	600+	600+	600+
GLSC-1	11	600+	600+	600+	600+	600+	600+	600+	600+	600+	600+	600+	600+	600+	600+	600+	600+	600+	600+
GLSC-2	7	600+	600+	600+	--	--	--	--	--	--	--	600+	600+	600+	600+	600+	600+	600+	600+
GLSC-2	9	600+	600+	600+	--	--	--	--	--	--	600+	600+	600+	600+	600+	600+	600+	600+	600+
GLSC-2	11	600+	600+	600+	--	--	--	--	--	--	600+	600+	600+	600+	600+	600+	600+	600+	600+
Alkali-carbonate	7	49	48	49	--	--	--	--	--	--	34	38	36	45	39	42	53	39	46
Alkali-carbonate	9	55	45	50	--	--	--	--	--	--	54	48	51	47	40	44	41	40	41
Alkali-carbonate	11	103	105	104	--	--	--	--	--	--	31	33	32	33	36	35	37	29	33
Recycled	7	89	51	70	85	48	67	93	40	62	115	66	91	104	57	81	70	49	60
Recycled	9	85	42	64	90	32	61	85	38	62	107	55	81	71	45	58	75	43	59
Recycled	11	60	31	46	60	35	48	70	30	50	74	32	53	73	31	52	71	36	54

TABLE XXI
EXTENDED ROOM TEMPERATURE AGING OF HANDSHEETS CONTAINING REWETTING AGENT

Pulp Used	pH	Rewetting Agent, %	Aging Time											
			4 Hours			2 Weeks			4 Weeks			8 Weeks		
			Wire	Felt	Av.	Wire	Felt	Av.	Wire	Felt	Av.	Wire	Felt	Av.
NSSC	7	0.025	248	139	194	336	191	264	260	138	199	386	284	335
	7	0.05	170	79	125	249	167	208	208	120	164	168	169	169
	7	0.125	169	69	119	224	128	176	190	130	160	275	226	251
NSSC	11	0.025	108	55	82	301	265	283	344	224	284	326	289	308
	11	0.05	109	69	89	228	181	205	227	171	199	232	227	230
	11	0.125	77	49	63	127	95	111	212	109	161	109	103	106
GLSC-1	11	0.05	597+	399	498+	224	160	192	493	224	359	313	228	271
	11	0.075	271	214	243	238	136	187	236	320	278	212	220	216
	11	0.1	113	61	87	57	54	56	95	97	96	104	115	110
	11	0.125	36	28	32	32	37	35	40	36	38	39	35	37
GLSC-2	7	0.025	600+	600+	600+	--	--	--	600+	600+	600+	600+	600+	600+
	7	0.050	600+	600+	600+	--	--	--	600+	600+	600+	600+	600+	600+
	7	0.125	600+	600+	600+	--	--	--	--	--	--	600+	600+	600+
GLSC-2	11	0.05	600+	600+	600+	--	--	--	--	--	--	46	600+	323+
	11	0.125	555	570	563	--	--	--	--	--	--	24	518	270
	11	0.25	205	200	203	--	--	--	146	77	112	25	80	53
Alkali-carbonate	11	0.025	33	22	28	--	--	--	24	45	35	31	30	31
	11	0.05	29	21	25	--	--	--	32	34	33	28	37	28
	11	0.125	32	22	27	--	--	--	19	29	24	20	28	24
			12 Weeks			16 Weeks								
			Wire	Felt	Av.	Wire	Felt	Av.	Wire	Felt	Av.	Wire	Felt	Av.
			339	373	356	339	373	356	473	403	438	398	328	363
			418	430	424	418	430	424	398	328	363	398	328	363
			350	321	336	350	321	336	282	304	293	282	304	293
			368	237	303	368	237	303	315	221	268	315	221	268
			268	237	253	268	237	253	170	127	149	170	127	149
			119	101	110	119	101	110	124	91	108	124	91	108
			478	209	344	478	209	344	197	167	182	197	167	182
			101	112	107	101	112	107	173	127	150	173	127	150
			60	57	59	60	57	59	60	63	62	60	63	62
			43	29	36	43	29	36	40	40	35	40	40	35
			600+	600+	600+	600+	600+	600+	600+	600+	600+	600+	600+	600+
			600+	600+	600+	600+	600+	600+	600+	600+	600+	600+	600+	600+
			600+	600+	600+	600+	600+	600+	600+	600+	600+	600+	600+	600+
			37	39	38	37	39	38	415+	245+	330+	415+	245+	330+
			34	308	171	34	308	171	202	415+	309+	202	415+	309+
			189	94	142	189	94	142	205	83	144	205	83	144

TABLE XXII
THE EFFECT OF CALCIUM CHLORIDE^a ON THE WATER
ABSORBENCY OF HANDSHEETS

Pulp Used	pH	Through Penetration,			Waterdrop,		
		Wire	sec Felt	Av.	Wire	sec Felt	Av.
NSSC control	7	3	3	3	185	156	171
NSSC with CaCl ₂	7	1	1	1	42	43	43
NSSC control	11	3	2	3	188	155	172
NSSC with CaCl ₂	11	3	3	3	167	167	167
Alkali carbonate control	7	2	2	2	49	48	49
Alkali carbonate with CaCl ₂	7	1	1	1	28	28	28
Alkali carbonate control	11	2	2	2	103	105	104
Alkali carbonate with CaCl ₂	11	7	8	8	600+	600+	600+
GLSC-2 control	11	600+	600+	600+	600+	600+	600+
GLSC-2 with CaCl ₂	11	600+	600+	600+	600+	600+	600+
Recycled control	7	1	1	1	89	57	70
Recycled with CaCl ₂	7	1	1	1	57	56	57
Recycled control	11	1	1	1	60	31	46
Recycled with CaCl ₂	11	1	1	1	92	64	78

^a100 ppm of CaO added as CaCl₂.

Sheets were aged 4 hours at 23°C before measurements were made.

Acid treatments were intended to remove the adverse effects of magnesium and calcium ions on paper wettability. For this purpose, 100-g batches of the refined GLSC pulp were treated with HCl in a stainless steel container. Each batch of pulp was diluted to 20 liters with deionized water, and sufficient HCl was added to adjust the pH to 1.1. After being stirred slowly for one hour, the pulp was drained on filter paper. The pulp was then resuspended and filtered several times to remove the acid. Handsheets were prepared from this pulp at pH levels of 7, 9, and 11, and the sizing results are presented in Table XXIII.

TABLE XXIII
THE EFFECT OF ACID TREATMENT ON THE WATER ABSORBENCY
OF GLSC-1 HANDSHEETS

pH	Aging Time, hr	Aging Temperature											
		23°C						105°C					
		Through Penetration, sec			Waterdrop, sec			Through Penetration, sec			Waterdrop, sec		
		Wire	Felt	Av.	Wire	Felt	Av.	Wire	Felt	Av.	Wire	Felt	Av.
7	0.5	1	1	1	68	43	56	1	1	1	88	59	74
	1.0	1	1	1	80	48	64	1	1	1	109	85	97
	2.0	1	1	1	89	60	75	1	1	1	92	89	91
	4.0	1	1	1	82	59	71	1	1	1	96	88	92
9	0.5	1	1	1	196	181	189	1	1	1	263	178	221
	1.0	1	1	1	192	157	175	1	1	1	228	182	205
	2.0	1	1	1	180	170	175	1	1	1	281	190	236
	4.0	1	1	1	216	239	228	1	1	1	315	223	269
11	0.5	1	1	1	102	94	98	1	1	1	94	61	78
	1.0	1	1	1	100	87	94	1	1	1	96	62	79
	2.0	1	1	1	112	91	101	1	1	1	106	64	85
	4.0	1	1	1	102	88	96	1	1	1	100	62	81

The effect of 1, 5, and 10% Calgon on self-sizing in handsheets prepared from GLSC pulp was examined. Calgon was added as a 1% solution to 0.5% consistency pulp at pH 7. It was allowed to stir with the pulp for a minimum of five minutes before handsheets were prepared. The sizing values are listed in Table XXIV.

The effect of four hours contact time at 1 and 10% addition levels of Calgon to the pulp at pH 7 was also examined. Handsheets were prepared five minutes after addition of Calgon, and then another set was prepared four hours later in the same manner as previously described. Sizing results are given in Table XXV.

Four sets of handsheets were prepared to determine the effect of a combination of 1% Calgon and 0.05, 0.075, 0.10, and 0.125% of anionic rewetting agent on the wettability of handsheets. Calgon was added to the pH 7 pulp before the surfactant and each was stirred in a minimum of five minutes before preparation of handsheets. The size test results are given in Table XXVI.

TABLE XXIV
THE EFFECT OF CALGON ON THE WATER ABSORBENCY OF GLSC-1 HANDSHEETS

Calgon Added, %	Aging Time, hr	Aging Temperature									
		23°C					105°C				
		Through Penetration, sec		Waterdrop, sec		Wire	Through Penetration, sec		Waterdrop, sec		Wire
		Felt	Av.	Felt	Av.		Felt	Av.	Felt	Av.	
1	0.5	3	3	192	189	191	3	3	276	229	253
	1.0	3	3	223	211	217	3	3	384	296	340
	2.0	3	3	256	232	244	3	3	271	211	241
	4.0	3	3	255	224	240	3	3	293	217	255
5	0.5	3	3	335	234	285	3	3	353	270	312
	1.0	3	3	375	220	298	8	5	520	560	540
	2.0	3	3	367	232	300	12	5	600+	600+	600+
	4.0	3	3	387	235	311	34	21	600+	600+	600+
10	0.5	3	3	225	199	212	6	4	360	214	287
	1.0	3	3	213	188	201	18	10	600+	600+	600+
	2.0	3	3	265	191	228	18	12	600+	600+	600+
	4.0	3	3	265	226	246	30	25	600+	600+	600+

NOTE: Handsheets were prepared at pH 7.

TABLE XXV
THE EFFECT OF EXTENDED CONTACT TIME OF CALGON ON THE WATER
ABSORBENCY OF GLSC-1 HANDSHEETS

Calgon Added, %	Aging Time, hr	Aging Temperature											
		23° C					105° C						
		Through			Waterdrop, sec		Penetration, sec	Through			Waterdrop, sec		
		Wire	Felt	Av.	Wire	Felt		Av.	Wire	Felt	Av.	Wire	Felt
1.0	0.5	2	2	2	217	178	198	2	2	2	197	135	166
	1.0	2	2	2	232	183	208	2	2	2	217	157	187
	2.0	2	2	2	180	156	163	3	3	3	293	300	297
	4.0	2	2	2	197	181	189	13	11	12	600+	600+	600+
1.0 ^a	0.5	2	1	2	112	89	101	2	2	2	165	159	162
	1.0	2	1	2	136	86	111	2	2	2	267	197	232
	2.0	2	1	2	119	86	103	5	4	5	547	467	507
	4.0	2	1	2	122	106	116	7	6	7	600+	600+	600+
10.0	0.5	5	3	4	449	331	390	10	5	8	600+	489	545+
	1.0	4	4	4	441	290	366	14	9	12	600+	572+	586+
	2.0	4	3	4	427	291	359	16	16	16	600+	600+	600+
	4.0	4	3	4	446	261	354	22	20	21	600+	600+	600+
10.0 ^a	0.5	3	2	3	287	169	228	3	2	3	282	206	244
	1.0	3	2	3	292	167	230	3	2	3	310	206	258
	2.0	3	2	3	300	208	254	3	3	3	350	303	327
	4.0	3	2	3	313	208	261	5	3	4	480	324	402

^aCalgon stirred in furnish four hours before handsheets were prepared.

NOTE: Handsheets were prepared at pH 7.

TABLE XXVI
THE EFFECT OF CALGON PLUS ANIONIC REWETTING AGENT ON THE WATER
ABSORBENCY OF GLSC-1 HANDSHEETS

Aging Time, hr		Aging Temperature											
		23°C					105°C						
		Through		Waterdrop, sec		Through		Penetration, sec		Waterdrop, sec			
Additive	hr	Wire	Felt	Av.	Wire	Felt	Av.	Wire	Felt	Av.	Wire	Felt	Av.
1.0% Calgon + 0.050% rewetting agent	0.5	1	1	1	48	38	43	1	1	1	64	55	60
	1.0	1	1	1	47	47	47	2	2	2	98	70	84
	2.0	1	1	1	53	40	47	4	4	4	223	185	204
	4.0	1	1	1	50	38	44	11	7	9	600+	600+	600+
1.0% Calgon + 0.075% rewetting agent	0.5	1	1	1	30	21	26	1	1	1	36	34	35
	1.0	1	1	1	34	31	33	1	1	1	46	40	43
	2.0	1	1	1	42	35	39	1	1	1	72	60	66
	4.0	1	1	1	41	37	39	7	8	8	510	600+	545+
1.0% Calgon + 0.1% rewetting agent	0.5	1	1	1	27	20	24	1	1	1	38	36	37
	1.0	1	1	1	38	38	38	1	1	1	39	42	41
	2.0	1	1	1	35	32	34	1	1	1	58	58	58
	4.0	1	1	1	33	34	34	4	4	4	246	227	237
1.0% Calgon + 0.125% rewetting agent	0.5	1	1	1	27	28	28	1	1	1	39	38	39
	1.0	1	1	1	36	35	36	1	1	1	46	41	44
	2.0	1	1	1	36	31	34	2	2	2	83	85	84
	4.0	1	1	1	39	37	38	4	4	4	218	217	218

NOTE: Handsheets were prepared at pH 7.

The effect of Versene 100 on the wettability of GLSC handsheets was also examined. In this series of experiments, handsheets were prepared at pH 5 and 9 incorporating 0.4-10.0% of Versene 100. The Versene was added as a 10% solution to the 0.5% pulp slurry. Sizing results are listed in Table XXVII.

The final series of handsheets was prepared to determine the effect of pulp temperature at the time of handsheet formation on the wettability of the handsheets. Handsheets were prepared over a temperature range that may be found in commercial papermaking and somewhat above. The temperatures chosen were 7, 23, 60, and 90°C. Handsheets were prepared at pH 7 with and without the addition of 0.1% of the anionic and two nonionic rewetting agents. For each set of sheets, the 0.5% pulp in the stainless steel container and the dilution water were maintained at the same temperature. Sizing results are recorded in Table XXVIII.

As a follow-up to this work, the solvent extractables content of the handsheets formed in the above temperature study (in the absence of rewetting agents) was determined by extraction with 2:1 benzene-alcohol. The results are recorded in Table XXIX.

TABLE XXVII
THE EFFECT OF VERSENE 100 ON THE WATER ABSORBENCY OF GLSC-1 HANDSHEETS

Versene 100 Added, %	pH	Aging Time, hr	Aging Temperature											
			23°C					105°C						
			Through			Penetration, sec			Through			Penetration, sec		
			Wire	Felt	Av.	Wire	Felt	Av.	Wire	Felt	Av.	Wire	Felt	Av.
0.4	5	0.5	1	1	1	145	74	110	2	2	2	187	163	175
		1.0	1	1	1	145	94	119	2	2	2	192	109	151
		2.0	1	1	1	142	100	121	2	2	2	239	285	262
		4.0	1	1	1	140	105	122	10	9	10	600+	600+	600+
0.4	9	0.5	1	1	1	185	150	168	3	1	2	454	151	303
		1.0	1	1	1	190	146	168	3	4	4	382	464	423
		2.0	1	1	1	157	139	148	6	9	8	600+	600+	600+
		4.0	1	1	1	181	160	171	12	16	14	600+	600+	600+
0.8	5	0.5	1	1	1	131	103	117	1	1	1	161	168	165
		1.0	1	1	1	178	169	174	2	2	2	231	262	247
		2.0	1	1	1	199	159	179	2	2	2	285	279	282
		4.0	1	1	1	196	152	174	5	4	5	510	370	440
0.8 ^a	5	0.5	1	1	1	109	66	88	1	1	1	128	103	116
		1.0	1	1	1	124	89	107	1	1	1	105	95	100
		2.0	1	1	1	122	111	117	1	1	1	114	103	109
		4.0	1	1	1	128	108	118	1	1	1	137	126	122
10.0 ^b	5	0.5	2	2	2	133	108	121	2	2	2	148	102	125
		1.0	2	2	2	143	164	154	2	2	2	147	118	133
		2.0	2	2	2	160	155	158	4	3	4	295	233	264
		4.0	2	2	2	150	117	134	4	3	4	306	299	303
10.0 ^{ab}	5	0.5	2	2	2	115	148	132	2	2	2	171	137	154
		1.0	2	2	2	159	106	133	2	2	2	162	126	143
		2.0	2	2	2	122	113	118	2	2	2	150	155	153
		4.0	2	2	2	153	137	145	2	2	2	163	165	164

^aversene stirred in furnish four hours before handsheets were prepared.
^bversene present in dilution water.

TABLE XXVIII

THE EFFECT OF VARIATIONS IN FURNISH TEMPERATURE ON THE WATER ABSORBENCY OF GLSC HANDSHEETS

Temp., °C	Additive	Aging Time, hr	Aging Temperature											
			23°C						105°C					
			Through Penetration, sec			Waterdrop, sec			Through Penetration, sec			Waterdrop, sec		
			Wire	Felt	Av.	Wire	Felt	Av.	Wire	Felt	Av.	Wire	Felt	Av.
7	--	0.5	2	2	2	127	131	129	2	2	2	247	184	216
		1.0	2	2	2	151	132	142	4	3	4	407	330	369
		2.0	2	2	2	143	124	134	22	21	22	600+	600+	600+
		4.0	2	2	2	142	121	132	600+	600+	600+	600+	600+	600+
23	--	0.5	2	2	2	97	92	95	3	3	3	290	233	262
		1.0	2	2	2	81	107	94	5	5	5	478	536	507
		2.0	2	2	2	90	85	88	22	19	21	600+	600+	600+
		4.0	2	2	2	110	96	103	600+	600+	600+	600+	600+	600+
60	--	0.5	1	1	1	55	58	57	1	1	1	74	76	75
		1.0	1	1	1	48	41	45	2	2	2	192	146	169
		2.0	1	1	1	42	47	45	5	5	5	376	360	368
		4.0	1	1	1	47	45	46	55	58	57	600+	600+	600+
90	--	0.5	1	1	1	17	14	16	1	1	1	16	15	16
		1.0	1	1	1	15	14	15	1	1	1	16	17	17
		2.0	1	1	1	14	13	14	1	1	1	18	18	18
		4.0	1	1	1	14	12	13	1	1	1	46	38	42
7	0.1% Anionic rewetting agent	0.5	1	1	1	19	14	17	1	1	1	30	27	29
		1.0	1	1	1	17	15	16	1	1	1	46	37	42
		2.0	1	1	1	17	15	16	2	2	2	76	67	72
		4.0	1	1	1	17	19	18	16	16	16	590+	600+	595+
23	0.1% Anionic rewetting agent	0.5	1	1	1	22	21	22	1	1	1	31	28	30
		1.0	1	1	1	24	26	25	1	1	1	32	35	33
		2.0	1	1	1	26	26	26	2	2	2	73	67	70
		4.0	1	1	1	33	26	30	4	4	4	341	353	347
60	0.1% Anionic rewetting agent	0.5	1	1	1	37	34	36	1	1	1	38	31	35
		1.0	1	1	1	35	32	34	1	1	1	47	45	46
		2.0	1	1	1	34	31	33	2	2	2	187	157	172
		4.0	1	1	1	32	27	30	23	19	21	600+	600+	600+
7	0.1% Nonionic rewetting agent-1	0.5	1	1	1	18	14	16	1	1	1	15	19	17
		1.0	1	1	1	25	21	23	1	1	1	26	23	25
		2.0	1	1	1	21	17	19	1	1	1	46	44	45
		4.0	1	1	1	23	17	20	1	1	1	60	54	57
23	0.1% Nonionic rewetting agent-1	0.5	1	1	1	20	21	21	1	1	1	27	23	25
		1.0	1	1	1	16	20	18	1	1	1	29	29	29
		2.0	1	1	1	18	19	19	1	1	1	37	38	38
		4.0	1	1	1	18	19	19	1	1	1	60	59	60
60	0.1% Nonionic rewetting agent-1	0.5	1	1	1	16	17	17	1	1	1	19	17	18
		1.0	1	1	1	16	18	17	1	1	1	22	19	21
		2.0	1	1	1	17	17	17	1	1	1	22	18	20
		4.0	1	1	1	17	17	17	1	1	1	43	42	43
7	0.1% Nonionic rewetting agent-2	0.5	1	1	1	22	17	20	1	1	1	20	16	18
		1.0	1	1	1	21	19	20	1	1	1	27	24	26
		2.0	1	1	1	21	19	20	1	1	1	43	40	43
		4.0	1	1	1	23	19	21	1	1	1	92	92	92
23	0.1% Nonionic rewetting agent-2	0.5	1	1	1	21	19	20	1	1	1	20	14	14
		1.0	1	1	1	22	23	23	1	1	1	26	20	20
		2.0	1	1	1	21	19	20	1	1	1	30	29	29
		4.0	1	1	1	19	19	19	1	1	1	73	76	76
60	0.1% Nonionic rewetting agent-2	0.5	1	1	1	23	22	23	1	1	1	26	26	26
		1.0	1	1	1	23	22	23	1	1	1	31	31	31
		2.0	1	1	1	23	22	23	1	1	1	52	58	55
		4.0	1	1	1	26	24	25	1	1	1	115	139	127

NOTE: Handsheets were prepared at pH 7.

TABLE XXIX

BENZENE-ALCOHOL EXTRACTIVES OF GLSC-1 HANDSHEETS PREPARED
AT VARIOUS TEMPERATURES

Temperature of Handsheet Preparation, °C	Total Extractives, %
7	3.58
23	3.63
60	3.72
90	2.84

NOTE: Handsheets were prepared at pH 7.
The extractions were carried out for 8 hours.

DISCUSSION

Results obtained in the pulp processing and analysis portion of the program (Tables II-VI) revealed several noteworthy effects. The metal (inorganic) composition of the pulps differed quite markedly (Table III). Of particular interest to self-sizing are the high calcium and magnesium levels in the green liquor pulps. The conventional NSSC pulp also had a high calcium level, but this was coupled with a high sodium content. The recycled fiber pulp contained high levels of silicon, aluminum, iron, calcium, and titanium. Most of these components were present in the secondary fiber before repulping and may not be bound in the same manner as in the virgin pulps. In general, secondary fiber pulps do not present serious self-sizing problems if the organic binders are removed in the pulping and cleaning operations.

In general, high levels of magnesium and calcium can lead to the formation of insoluble salts with the natural and fatty resin acids present in the pulp, depending on the pH. In contrast, high levels of sodium would be expected to reduce or minimize self-sizing at high pH through the formation of water-soluble soaps. These soaps, if present at sufficiently high concentrations, would form micelles which could aid in removing unsaponifiable resinous components during the washing cycles.

With respect to the organic solvent extractables (Table IV), the green liquor pulps contained high levels of extractables before and after classification, suggesting that a considerable amount of natural resins remained with the whole fibers. The green liquor pulps also contained somewhat higher levels of free acids, whereas the recycled pulp contained relatively high levels of neutrals and unsaponifiables. The latter may be due in part to the retention of adhesive components in the repulping operation. Swanson and Cordingly (1) showed that stearic acid

volatilizes at room temperature from the crystalline state and becomes adsorbed on cellulose surfaces. In the presence of calcium and magnesium ions, insoluble salts of fatty acids may form. These salts provide a more stable sizing condition, since overturning of the fatty molecules is reduced or prevented.

Handsheets formed from the various pulps had rough surfaces before and after solvent extraction or fiber classification (Table VI). Greater differences existed in porosity where, as would be expected, removal of the fines fraction (classification) greatly increased porosity. Aside from this effect, GLSC-2 showed the lowest porosity and the alkali carbonate pulp the highest porosity. Porosity is an important parameter in the penetration of liquids into porous media, as indicated by the Washburn equation:

$$\frac{d\ell}{dt} = \frac{\gamma_L r \cos \theta}{4\eta \ell} \quad (1)$$

where $\frac{d\ell}{dt}$ is the rate of penetration, γ_L is the surface tension of the liquid, r is the pore radius, η is the viscosity of the liquid, and θ is the contact angle formed by the liquid on the solid surface. Equation (1) is for an idealized system based on uniform cylindrical pores.

The effect of surface roughness is indicated by the modified Young's equation:

$$\cos \theta_{AA} = \frac{(\gamma_S - \gamma_{SL})}{\gamma_L} \sigma = \sigma \cos \theta_A \quad (2)$$

where θ_{AA} is the apparent advancing contact angle, γ_S is the surface tension of the solid, γ_{SL} is the interfacial tension, θ_A is the real advancing contact angle, and σ

is Wenzel's roughness factor, which is equal to the $\frac{\text{actual surface area}}{\text{geometric surface area}}$. Hence, roughness may either increase or decrease wetting and penetration depending upon whether the contact angle is acute or obtuse.

Water absorbency in sheets formed from conventional NSSC pulp (Table VII, Fig. 1) showed a marked increase in self-sizing as a function of aging time, particularly among the oven-aged samples at pH levels of 7 and 9, whereas relatively minor increases were evident at 23°C. A consistent pattern with respect to pH was not evident under the two aging temperatures.

Comparable data for the green liquor pulps are presented in Tables VIII and IX. These pulps rapidly developed a high level of self-sizing. In fact, waterdrop values of 600+ seconds were obtained in all oven-aged samples, regardless of pH. Aging at room temperature produced similar results in GLSC-2 and rapidly increased sizing in GLSC-1, as shown in Fig. 2. The rapid development of water resistance was not expected at pH 11, in which case the presence of water-soluble soaps would normally assist wetting and penetration.

Table X and Fig. 3 show the effects of aging on the development of self-sizing in the AC pulp. In this case, waterdrop values reached 600+ seconds after two hours of aging at 105°C, regardless of pH, but aging at room temperature had little effect on water absorbency. The effect of pH was consistent at both aging temperatures but, once again, the pH 11 condition proved most adverse to water absorptivity (Fig. 3).

Results obtained with the recycled fiber pulp (Table XI, Fig. 4) showed a rather slow increase in water resistance as a function of oven aging, and the levels attained were well below those in the other pulps, regardless of pH. Aging at room

temperature produced low sizing values roughly comparable to those of the AC pulp. As in the previous case, waterdrop levels remained essentially unchanged as a function of room-temperature aging time. As previously indicated, recycled fiber pulps would not be expected to produce high levels of self-sizing, since most residual resinous material is removed in the repulping operation.

The effects of adding a commercial anionic rewetting agent to the NSSC, GLSC, and AC pulp are shown in Tables XII to XV. The effects of the rewetting agent under highly sized (adverse) conditions are shown in Fig. 5 and 6. Addition of 0.05-0.125% rewetting agent to the conventional NSSC pulp provided some advantage at room temperature, particularly at pH 7 (Table XII). Similar effects were obtained at 0.125% addition after aging at 105°C. Less favorable results were obtained at pH 11 after oven aging (Table XIII). Results in Fig. 5 show the advantage for rewetting agent addition in excess of 0.05%. Some advantage is also indicated at pH 11 over pH 7 at room temperature for all surfactant additions.

The rewetting agent produced improved water absorbency in the GLSC-1 pulp at 23°C at all addition levels and at additions greater than 0.05% at 105°C (Table XIV). However, it is evident in Fig. 6 that surfactant additions greater than 0.1% would be required for high water absorbency.

Results in Table XV and Fig. 7 show that addition of the rewetting agent to the AC pulp improved water absorbency somewhat but had no effect on GLSC-2 at pH 7 and excessively high additions would be required at pH 11.

Classification of the various pulps (Tables XVI and XVII) reduced self-sizing to some extent in all pulps except GLSC-2 (compare results in Fig. 8-11). This is tentatively assumed due to the fact that GLSC-2 contained the highest levels

of solvent extractables, free acids, and unsaponifiabiles after classification. The combination of classification and solvent extraction essentially eliminated all evidence of self-sizing (Table XVIII). This, of course, satisfies scientific curiosity but does not represent a viable solution to the self-sizing problem.

Hot water extraction (Table XIX) greatly reduced self-sizing in GLSC-1 and the AC pulps but was less effective in the GLSC-2 pulp. Reasons for the difference in the effectiveness of hot water extraction between GLSC-1 and GLSC-2 are not apparent, but it is assumed to be related to the total solvent extractables listed in Table IV.

Extended room temperature aging (Table XX) produced high levels of self-sizing in the NSSC pulp at pH 9 but to a lesser extent at pH 7 and 11. The GLSC pulps were previously found to become rapidly self-sized, so extended aging did not produce any changes. The AC and recycled fiber pulps showed little change with extended room-temperature aging, both remaining at low sizing levels.

Results obtained in aging handsheets for extended periods in the presence of the anionic wetting agent are presented in Table XXI. The presence of rewetting agent proved to be of little or no benefit in the NSSC and AC pulps, but some advantage was obtained with the GLSC pulps at pH 11, particularly in the case of GLSC-1.

The influence of calcium chloride on water absorption is presented in Table XXII. The effect of the calcium salt depended on pH. Addition of the salt at pH 7 tended to reduce self-sizing, whereas a detrimental effect is indicated at pH 11, most notably in the AC pulp. These results are somewhat puzzling, since formation of troublesome insoluble calcium salts of the natural resinous components would be expected to occur in the pH range of 6-8 rather than at pH 11.

The remainder of the program was directed to alleviating or eliminating the development of self-sizing in GLSC pulp, which was indicated in the preceding sections to be the most troublesome. GLSC-1 was selected for this work primarily on the basis of its extremely high calcium content. The water absorption properties of this pulp are presented in Table VIII, and comparisons may be made with these results in much of the work that follows.

Treatment of the GLSC pulp with acid (HCl) prior to sheet formation produced very significant reductions in self-sizing, presumably through removal of calcium and magnesium ions (Table XXIII). This was supported by ash contents of 1.44% for untreated pulp compared with 0.19% for the acid-treated pulp.

The effectiveness of sequestering and chelating agents in reducing self-sizing by tying up or removing calcium ions is presented in Tables XXIV-XXVII. The presence of Calgon reduced the self-sizing effect in room temperature aged sheets at the 1% Calgon addition level, but little or no additional benefit was derived from 5 and 10% Calgon additions (Table XXIV). Increasing the contact time of Calgon with the pulp to four hours (Table XXV) produced little benefit in water absorptivity at the 1% Calgon level. More substantial reductions in sizing were obtained at the 10% Calgon addition level, but the waterdrop numbers remained relatively high and the addition level was excessive. Combinations of 1% Calgon and 0.5-0.125% addition of the anionic rewetting agent were examined in the next series of tests (Table XXVI). These results show that the combination of 1% of Calgon plus 0.1 or 0.125% of rewetting agent was moderately effective in reducing self-sizing although, once again, waterdrop numbers remained relatively high after oven aging. It would

appear, however, that combination of 1% Calgon plus rewetting agent was more effective than Calgon alone or extended contact with Calgon.

The effectiveness of Versene as a chelating agent is shown in Table XXVII. Where direct comparison with previous data can be made (pH 9), it would appear that 0.4% of Versene 100 produced some improvements in water absorptivity at 23°C, but little advantage was evident after oven aging. Addition of 0.8% of Versene at pH 5 effected further reductions in waterdrop values, but once again the papers retained a significant level of sizing.

The final series of handsheets examined the effect of pulp temperature during sheet formation at pH 7 in the absence and presence of the previously used anionic rewetting agent and two nonionics (Table XXVIII). While the trends in water absorbency are not necessarily consistent, the results indicate that increasing the forming temperature to 90°C in the absence of surfactant essentially eliminated self-sizing. This, of course, is an unrealistic temperature, although temperatures of this magnitude are reached in the deinking of nongroundwood broke, in which case the pulp is washed and cleaned after the cooking stage. Actually, the results for the sheets formed at 90°C in Table XXVIII compare favorably with those formed at pH 11 from the hot water extracted pulp (Table XIX). However, emphasis is placed on the lower, more practical temperatures. For example, 0.1% of the nonionic surfactants produced substantially improved water absorbency at 23 and 60°C, and these are among the most encouraging results obtained in this study. Conceivably, slightly higher levels of the surfactant or longer contact time would further improve water absorption.

The final table (Table XXIX) lists the solvent extractables content of the handsheets prepared in the controlled temperature experiments in the absence of

rewetting agent. The extractables were essentially constant at the first three forming temperatures but then declined 0.8 or 0.9% at the highest temperature, under which condition self-sizing was essentially absent. It would be desirable to have available chemical analysis of the components removed at the 60 and 90-degree treatments, since that information may provide the answer to the self-sizing problem in green liquor pulps and possibly in other pulps as well.

CONCLUSIONS

The following conclusions are drawn on the basis of the evidence provided in this study:

1. Self-sizing develops to some degree as a function of aging in the four major types of pulp used in the production of corrugating medium, although to a very minor degree in recycled fiber pulp. The order of increasing sizing (reduced water absorbency) among the pulps tested was as follows:
 - a. recycled fiber
 - b. AC
 - c. conventional NSSC
 - d. green liquor
2. With the exception of the AC pulp, pH levels in the range of 7-11 had little or no consistent effect on self-sizing.
3. Rewetting agents were more effective in reducing self-sizing in some pulps than in others. The recycled fiber pulp did not require a rewetting agent, and the green liquor pulps proved most resistant to the beneficial effects of an anionic surfactant.
4. Classification (fines removal) reduced self-sizing in most pulps but not in one GLSC.

5. Addition of sequestering or chelating agents to GLSC pulp improved water absorbency to some extent but did not eliminate self-sizing.
6. Hot water extraction or high temperature forming greatly reduced self-sizing in GLSC pulp.
7. Forming sheets at 90°C essentially eliminated self-sizing in GLSC pulp as did the combination of classification and solvent extractions.
8. The most practical solution to the self-sizing problem appears to be the addition of 0.1% rewetting agent coupled with moderately high temperature forming conditions.

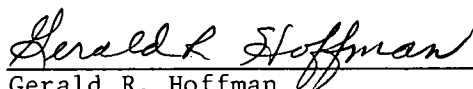
ACKNOWLEDGMENTS

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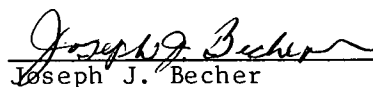
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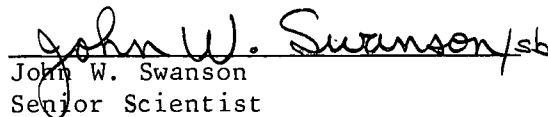
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APPENDIX I
CORRUGATING MEDIUM SELF-SIZING QUESTIONNAIRE

1. Company _____
2. Plant location _____
3. Is your plant experiencing a corrugating medium problem considered to be directly or indirectly related to self-sizing?
Yes _____ No _____ Don't know _____
4. Has your plant experienced a problem with self-sized medium in the past?
Yes _____ No _____ Don't know _____
5. Have you correlated the self-sized medium with the type of pulp stock?
Yes _____ No _____ Don't know _____

If so, check which type(s):

- a. Conventional semichemical pulps _____
 - b. Green liquor pulps _____
 - c. Recycled fiber pulps _____
 - d. Other (please specify) _____
6. How does/did the problem using self-sized corrugating medium manifest itself? Please check where appropriate.
- a. Poor fluting (highs and lows) _____
 - b. Poor adhesion at the single facer _____
Green bond strength _____ Final bond strength _____
 - c. Poor adhesion at the double backer _____
Green bond strength _____ Final bond strength _____
 - d. Other (please specify) _____
7. What corrugating variables are altered to accommodate self-sized medium? Please check where appropriate.
- a. Reduced corrugating machine speed _____
 - b. Preheating the medium _____
Lower temperature _____ Higher temperature _____
 - c. Steam shower pressures (temperature) _____
Reduced pressure _____ Increased pressure _____
 - d. Adhesive consumption _____
Increased _____ Decreased _____
 - e. Change of adhesive formulation _____. How? _____
 - f. Increased pressure roll pressure _____ Decreased pressure _____
 - g. Other (please specify) _____

8. What tests are regularly conducted on the corrugating medium to predetermine if self-sizing will cause problems in the converting process?

- a. Water drop tests _____
- b. Other liquid penetration tests (please specify) _____
- c. Adhesion tests (please specify) _____
- d. Other (please specify) _____

9. Please indicate the acceptable range of values for tests in Item 8.

<u>Test</u>	<u>Acceptable Range</u>	<u>Sample Age at Test</u>
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10. What remedial measures are taken to prevent self-sizing?

- a. Modification of the pulping process (specify) _____
- b. Addition of rewetting agents: _____
Anionic _____ Nonionic _____ Cationic _____
- c. Web cooled after driers _____
- d. Reduced stock inventory times _____
- e. Other (please specify) _____

11. What are the approximate costs of the remedial treatments in terms of dollars/year for all company operations? _____

12. Should the Institute pursue solution or control measures of the corrugating medium self-sizing problems? Yes _____ No _____ Don't know _____

13. Comments: _____

Signature _____ Title _____ Date _____

Please return the completed questionnaire in the postpaid envelope to:

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